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Surface Treatment of PTFE Polymer using RF-Plasma

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ABSTRACT

The surface of Teflon (poly-tetra-fluoroethylene –PTFE) films were treated by argon RF- plasma. Scanning electron microscope (SEM), atomic force microscopy (ATM) and X-Ray Diffraction (XRD) measurements was used to investigate and characterize surface treatment of the polymer used. The RF power input and the treatment time influence the chemical/morphological characteristics and the wettability of the plasma-treated samples. SEM measurements shows that, the unevenness disappears due to the grafted polymer on the surface and the surface is almost flat. The roughness of the PTFE surfaces increase with increases the treatment time; this is due to the bombardment of plasma species on the surface of PTFE films, hence it can support the adhesion improvement. The XRD patterns are characterized by the appearance of two peaks the dominant one in49.72 and small peak at 17.48 degrees in 2 theta axis. The most intense and sharp peak (at 49.72 in the 2 theta axis) indicates that the crystalline phase is the dominant phase in the treatment PTFE polymer. It is found that the plasma treatment modifies the polymer surface in both morphology and composition. Results show that the surface wettability is significantly enhanced after plasma treatment It is found that the plasma treatment modifies the polymer surface in both morphology and composition. Results show that the surface wettability is significantly enhanced after plasma treatment. It is found that, the change in surface properties depends on the treatment time. The higher the treatment time, the better the polymer treatment until a maximum occur at treatment time (t = 10 min.).

INTRODUCTION

Plasma treatment has an explosive increase in interest and use in industrial applications as for example in medical, biomedical, automobile, electronics, semiconductor and textile industry (Liu, X.M., *et al*., 2007; Morra, M., *et al*., 1990; Rossini, P., *et al*., 2003). A lot of intensive basic research has been performed in the last years, also in the field of textiles and technical textiles. This has resulted in an increasing knowledge of the possibilities of this process regarding the demands as wettability, shrinkage resistance of wool, dyeability, printability, coating and washability of conventional and technical textile. All day problems of wettability and adhesion, together with the environmental driven forces have increased the interest of industry today (Ionita, M.D., *et al*., 2010).

Activation is the bounding of hydroxyl, carbonyl and carboxylic acid groups to the surface. Weak bonds are broken and replaced by stable functional groups with a positive influence on adhesion and hydrophilisation. Reason is the increase of the surface energy. For polymers this surface energy is typical low which is the basis of the poor adhesion properties of such surfaces.

Among others materials, Teflon (PTFE - polytetrafluoroethylene) is extensively used in biomedical applications like construction of heart stents, vascular grafting, etc., because of its special properties as high thermal stability, chemical inertness, low surface tension and low coefficient of friction (Liu, X.M., *et al*., 2007). For some particular applications, it is necessary to improve the adhesion and wettability of the PTFE surface. As

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an example, it is well known that the usability, topography and surface chemistry can affect cell response on the surface (Favia, P., *et al*., 2006). Due to the ability to modify the surface characteristics without affecting the bulk material properties, radio frequency plasma sources are widely used for treatments of polymers (Ionita, E.R., *et al*., 2009).

Stefano Zanini *et al*. (2014) Show that the effects of O2 plasma treatments on poly (tetrafluoroethylene) (PTFE) sheets are deeply investigated. The chemical modifications owing to the plasma treatment are studied by means of attenuated total reflectance Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy (XPS), while the surface topography of the plasma-treated samples are assessed by atomic force microscopy (AFM) analyses. Finally, the ageing of the plasma-treated surfaces is investigated. The RF power input and the treatment time influence the chemical/morphological characteristics and the wettability of the plasma-treated samples. PTFE samples treated at low-power input (up to 50 W) is more hydrophilic than the untreated one. The AFM analysis shows a strong increase in the surface roughness as a consequence of the differential etching of the PTFE surface for samples treated at power inputs ≥100 W, with formation of globular structures.

The aim of the present work is to treat and/or modify the PTFE polymer using RF plasma source. Results on a factorial design experimentation for the most important plasma parameters; treatment time, gas pressure, RF power will be discussed.

Experimental Setup:

The discharge plasma was formed in parallel plate configuration of stainless-steel 304 electrodes with a diameter of 5 cm and spacing 6 cm, housed in a cylindrical stainless-steel vacuum chamber of 25 cm in diameter and height. The side and back of the two electrodes were covered with ceramic casing to prevent additional discharge. The lower electrode was powered by rf source (13.56 MHz and 0-200 W power), type ENI model OEM-6, through a matching box, while the upper electrode as well as the stainless-steel discharge chamber were grounded. The chamber was pumped down by a vacuum system to base pressure of 10−4 torr.

During all measurements, continues flow of a gas through the discharge chamber was maintained. High purity Ar gas was used as working gas and was fed to the chamber through a needle valve. The pressure of the working gas was varied between 1 − 10 torr and measured using a digital vacuum gauge (VAP 5).

Commercial flat sheets of PTFE 127 μm in thickness were purchased from

DuPont (USA). The gas used for plasma treatments was argon gas.

Surface characterization of PTFE polymer was conducted 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).

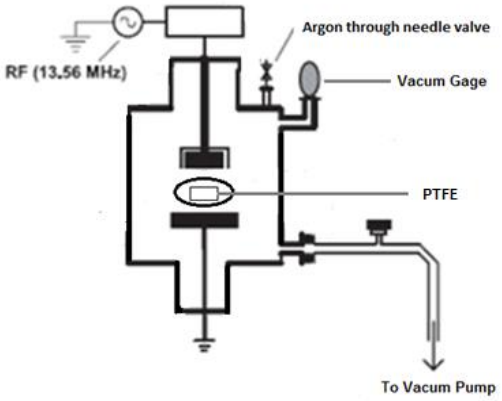

Fig. 1: shows a schematic drawing of the experimental set up.

RESULTS AND DISCUSSION

PTFE is one of the most inert polymer materials. The process goal here is the activation and/or modification of the PTFE surface.

3-1 SEM Observations:

Figures 2 (a)–(d) show the surfaces of the PTFE films as observed using the SEM. Figure 2 (a) shows the surface of the untreated PTFE film. Some granular unevenness is observed because the film is originally prepared from PTFE powder.

Figure 2 (b) shows the surface of the PTFE film treated by the RF plasma at 100 W powers. In this photograph, the unevenness disappears due to the grafted polymer on the surface and the surface is almost flat.

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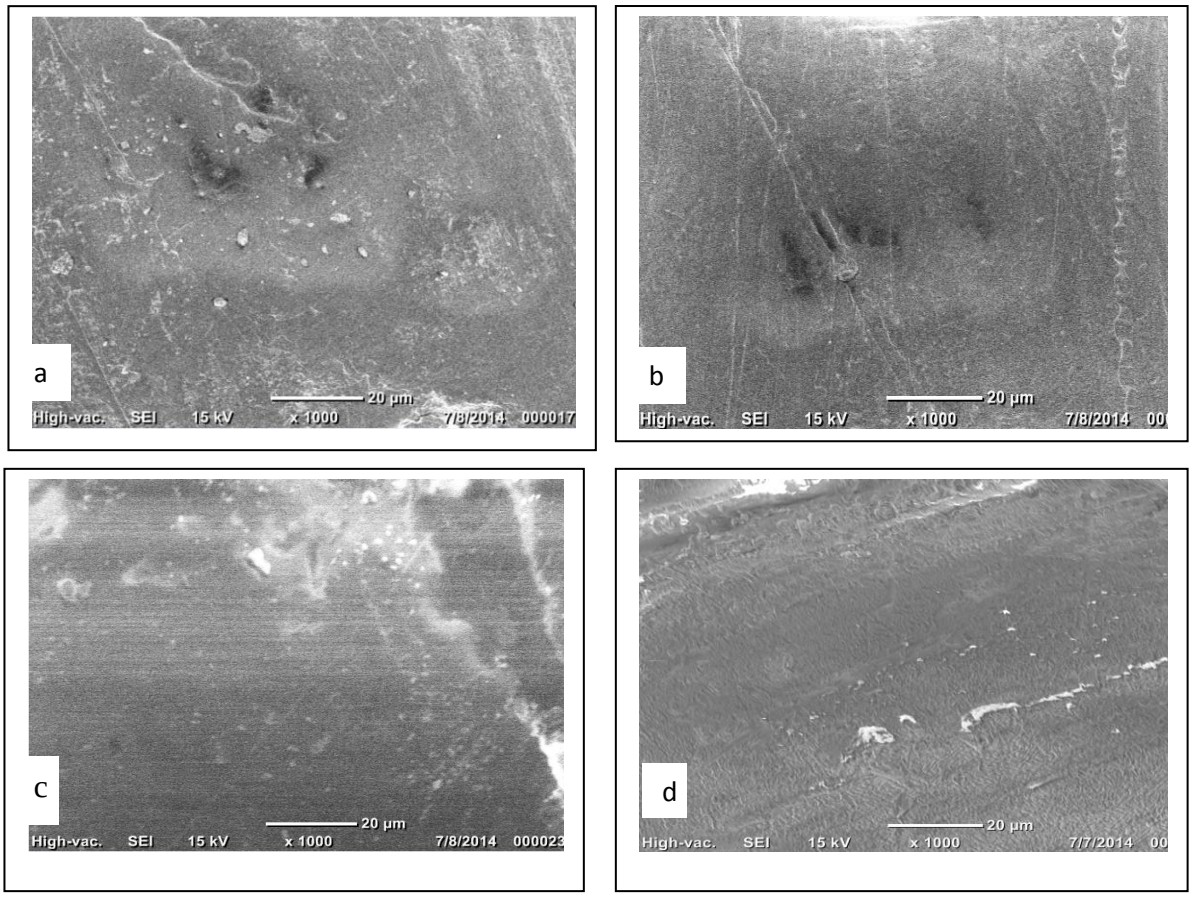
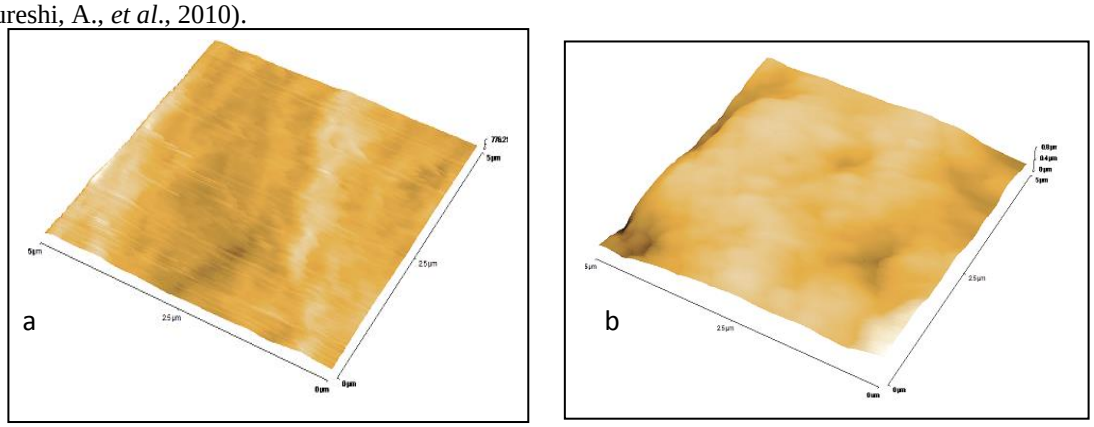


Fig. 2: (a,b, c and d) SEM images surface of PTFE film treatment with RFPlasma at 100 Watt power (a) untreated (b) 1 min treatment (c) 5 min treatment and (d) 20 min treatment at 1 torr gas pressure.

Since PTFE is a chemically inert polymer, plasma treatment has little effect on surface activation i.e. increase in surface energy. The increase in surface free energy is attributed to the functional utilization of the polymer surface with hydrophilic groups on the surface. There is a little increase in the polar component after all subsequent treatments, whereas remarkable change occurred in the dispersion component. This slight change in polar component is due to the chemically inert nature of PTFE. Important information obtained from the surface energy measurement is that the increase in polar component indicates the formation of covalent bonds (Pelagade, S.M., 2012).

3-2 Atomic Force Microscopy (AFM):

The surface morphology of argon treated PTFE samples were measured by AFM in contact mode on the 5 × 5 µm2 areas and are shown in Fig. 3 (a)-(d). Each AFM image was analyzed in terms of surface average roughness. The data show that, the average surface roughness (RMS) increase with treatment time. The average surface roughness (RMS) for untreated film and argon plasma treated film for 20 min are 6.5 nm and 18.5 nm, respectively. The roughness of the PTFE surfaces increase with increases the treatment time; this is due to the bombardment of plasma species on the surface of PTFE films, hence it can support the adhesion improvement (Qureshi, A., *et al*., 2010).



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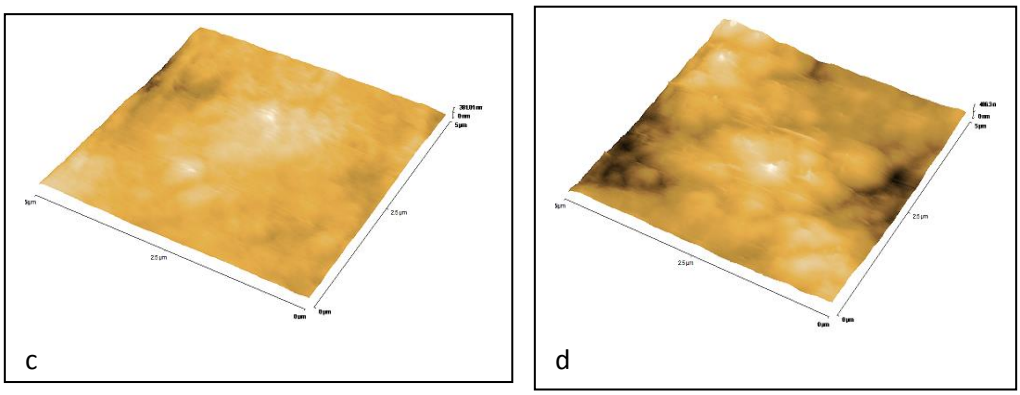

Fig. 3: (a,b, c and d) AFM images surface of PTFE film treatment with RF

Plasma at 100 watt power (a) untreated (b) 1 min treatment (c) 5 min treatment and (d) 20 min treatment at 1 torr gas pressure.

The increase in surface free energy indicates the improvement of adhesion on polymer surfaces. This is supported by AFM results as roughness increases.

Wilson *et al*. (2001) Show that the AFM of argon plasma treatment of PTFE resulted in a hummock relief and the RMS roughness being equal to 28 nm.

The increase in treatment times used for surface modification of polymers causes oxidation, removal of surface contaminants and improving of surface wettability. Long treatment time can cause chemical etching, increase in sample temperature, and creation of nanostructures and irreversible damage of the bulk properties. The main drawback of plasma treatment for activation of polymers is ageing. The various functional groups formed during plasma treatment are not stable with time. Hence, the surface tends to approach to its untreated state. Hence, the surface keeps losing its hydrophilic character spontaneously (Pelagade, S.M., 2012).

A corollary is that, when reporting on plasma-surface interaction, treatment time is very important parameter, since steady state is reached only quite some time after the beginning of the discharge (Morra, M., *et al*., 1990).

3-3 XRD measurements:

XRD measurements were performed on a Teflon sheet at different plasma treatment times to obtain information about the induced structural changes. Figure 4 (a-d) shows the obtained x-ray diffraction patterns for the Teflon samples at different treatment time and at RF plasma power = 100 watt. From these figures one can see that the peak intensity increases with increasing plasma treatment times. It's also shown that the XRD patterns are characterized by the appearance of two peaks the dominant one in49.72 and small peak at 17.48 degrees in 2 theta axis.

The small peak (at 17.48 in the 2 theta axis) is appeared sharply and increased by increasing the plasma treatment time. The most intense and sharp peak (at 49.72 in the 2 theta axis) indicates that the crystalline phase is the dominant phase in the treatment PTFE polymer.

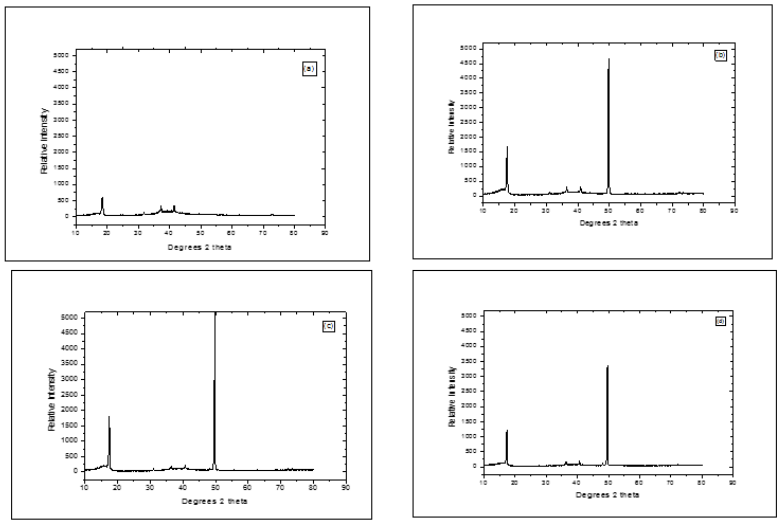

Fig. 4: X-ray diffraction patterns for PTFE film treatment with RF plasma at100 watt power (a) untreated (b) 1 min treatment (c) 5 min treatment and (d) 20 min treatment.

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Conclusion:

Surface modification by gas discharge plasmas plays a crucial role in the microelectronics industry. Exposure of polymers to suitable plasmas can cause chemical and physical changes to their surface or near-surface layers. These changes produce more reactive surfaces and affect wetting properties, cross-linking and molecular weight. Activation is the bounding of hydroxyl, carbonyl and carboxylic acid groups to the surface. Weak bonds are broken and replaced by stable functional groups with a positive influence on adhesion and hydrophilisation. Reason is the increase of the surface energy. For polymers this surface energy is typical low which is the basis of the poor adhesion properties of such surfaces. Cross linking is done with inert process gasses. Bondings are broken and will recombine, to form double or triple bonds or can form a bond with free radicals on another chain.

The present work deals with the treatment surface of polytetrafluoroethylene –PTFE film using RF glow discharge Argon plasma, chemical modification was dominant.

Commercial flat sheets of PTFE 127 μm in thickness were purchased from DuPont (USA). The gas used for plasma treatments was argon gas.

Surface characterization of PTFE polymer was conducted 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).

The PTFE surface is etched by ion bombardment during plasma discharge. It is worth noting that the etching rate depends closely on the composition of the process gas. In pure argon, the plasma-etched coating thickness in the PTFE ism proportional to the duration of treatment.

The modified surface was characterized using scanning electron microscopy (SEM), atomic force microscopy (AFM) and X-Ray Diffraction (XRD).

The SEM results show that increase in surface free energy is attributed to the functional utilization of the polymer surface with hydrophilic groups on the surface. There is a little increase in the polar component this indicates the formation of covalent bonds.

Each AFM image was analyzed in terms of surface average roughness. The data show that, the average surface roughness (RMS) increase with increasing the treatment time. PTFE samples treated at low-power input (up to 50 W) are more hydrophilic than the untreated one.

The study showed that the XRD patterns are characterized by the appearance of two peaks at 17.48 and 49.72 Degrees in 2 theta axisand the peak intensity increases with increasing plasma treatment times. The small peak (at 17.48 in the 2 theta axis) is appeared sharply and increased by increasing the plasma treatment time. The most intense and sharp peak (at 49.72 in the 2 theta axis) indicates that the crystalline phase is the dominant phase in the treatment PTFE polymer.

It is found that, the change in surface properties depends on the plasma treatment time. The higher the treatment time, the better the treatment until a critical treatment time is reached.

Therefore, one can conclude that, the system described in this paper provides an efficient and non-destructive means of altering the surface properties of polymer surfaces.

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