



## Research Article

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# Wear Properties of PTFE and C<sub>3</sub>F<sub>6</sub> Polymer Coated Carbon Steel Using RF Low-Pressure Plasma

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### Abstract

In this study, the surface of AISI 1050 carbon steel has been heat treated with different treatment parameters (fully softened, tempered) and a mechanical surface process (shot peening). Heat treated AISI 1050 carbon steel was coated in an RF vacuum plasma system with different plasma parameters (power, pressure, treatment time and type of gas). Two different coating processes were used: plasma grafting of sprayed polytetrafluoroethylene (PTFE) using argon and helium plasma and thin-layer deposition by hexafluoropropene (C<sub>3</sub>F<sub>6</sub>) plasma. The abrasion of coated surfaces was tested. After wear tests, surface morphology was analysed by scanning electron microscope (SEM) and optical microscope (OM). PTFE coating processes were found to be superior to C<sub>3</sub>F<sub>6</sub> plasma processes.

### Keywords

Polymers; Wear testing; Tribophysics; Surface analysis; Other surface engineering process

## Introduction

Polymer materials have been studied because of their favourable properties (such as low surface energy, good dielectric properties and good lubrication). These materials can be coated by various coating techniques [1,2]. Steel is used in engineering applications such as gear bearings, and steel components suffer from wear. Many investigations have focused on how to improve the wear life of steel using different coating methods.

Bearings, shafts, gears and bolts are examples of machine elements that are prone to wear. Erosion resistance, lubrication conditions and specific temperatures determine the behaviour of these elements from both economic and technological points of view. Research has focused on to how improve the wear life of machine elements. For this purpose, machine elements can be coated using different coating techniques, such as RF plasma sputtering and vacuum plasma [3]. One of the most important coating techniques is coating using polymers on metallic substrate. This technique has desirable properties and increases the wear life of the substrate [4].

Polytetrafluoroethylene (PTFE) is widely used in tribological material processing because it has a low friction coefficient. PTFE has a wide range of applications, including coating side plates, gears and plain bearings [5]. Furthermore, PTFE has good lubrication and dielectric properties [2,5]. PTFE reduces friction, wear and energy consumption of machine systems [6]. Another polymer that has desirable coating properties and is used to coat metallic components is hexafluoropropene (C<sub>3</sub>F<sub>6</sub>) [7]. In this study, PTFE and C<sub>3</sub>F<sub>6</sub> were coated on an AISI 1050 carbon steel substrate using low-pressure plasma discharge.

Recently used techniques to deposit PTFE and C<sub>3</sub>F<sub>6</sub> films on carbon steel employ plasma discharges. Understanding the effect of plasma parameters on the quality of the deposited thin film is important. Takashima and Oda studied the space and surface-charge behaviour of corona-charged PTFE thin films after plasma processing [8]. They examined the effect of an antistatic process using low-pressure discharge plasma and plasma and charge elimination processes by dipping in tap water. Their results show that the charge elimination of plasma processed samples improved independently of process gas type during the plasma processing. Caro et al. studied low-pressure plasma treatments of PTFE film using SO<sub>2</sub> process gas [9]. The variable plasma parameters were gas flow rate, power and treatment time. Results show that after plasma treatment an increase of the wettability occurred in terms of having the lowest static contact angle. Aderikha and Shapovalov studied the effect of RF plasma treatment on the structure and tribological behaviour of PTFE-polyoxadiazole fibre composites [10]. Results showed that fibre treatment raises the composite density, heat resistance and mechanical strength and improves the tribological properties. Dascalescu et al. studied engineered disk substrates (gray cast iron, sintered iron and Al390-T6) coated with PTFE/pyrrolidone (C1), PTFE/pyrrolidone (C2), PTFE/MoS<sub>2</sub> (C3) in a CO<sub>2</sub> environment [11]. Results show that substrate type had a major impact on the tribological performance of coatings. The coating of PTFE/MoS<sub>2</sub> was found to significantly improve the scuffing performance of Al390-T6 substrate. Furthermore, tribological performance was improved by increasing the CO<sub>2</sub> pressure. Demas and Polycarpou investigated the effect of the coating material on tribological properties [5]. Gray cast iron disks were coated with PTFE-based coatings, and the results showed that PTFE-based coatings had a low friction characteristic and high load-carrying capacity.

In the present study, a thin film of PTFE and C<sub>3</sub>F<sub>6</sub> is coated onto a heat treated AISI 1050 carbon steel sample using an RF vacuum plasma system. Another sample was surface treated mechanically by shot peening. The first sample was coated by plasma grafting of sprayed PTFE using argon and helium plasmas. The second sample was processed using thin layer deposition using C<sub>3</sub>F<sub>6</sub> plasma. The surfaces of the coated and non-coated samples were analysed by energy-dispersive X-ray spectroscopy (EDS), scanning electron microscope (SEM) and optical microscope (OM). Tribological properties of the coated surfaces were analysed by wear tests.

## Experimental Method

### Sample preparation: selection of coating materials and plasma parameters

AISI 1050 carbon steel (C-0.52%, Mn-0.75%, P-0.04%, S-0.05

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and the rest Fe) was used as a substrate. AISI 1050 plates (30 mm×30 mm×10 mm) were heat treated at different conditions and shot peened. Heat treatment parameters and average hardness values of substrates are given in Table 1.

PTFE and C<sub>3</sub>F<sub>6</sub> were used as coating materials. In this study, heat treated and shot peened AISI 1050 carbon steel was coated in an RF vacuum plasma system with different plasma parameters. The variable parameters were power, pressure, type of gas, and treatment time. AISI 1050 carbon steel surfaces were coated by plasma grafting of sprayed PTFE using argon and helium gases. A thin film layer of C<sub>3</sub>F<sub>6</sub> was deposited. Before and after the deposition process, surface morphology was analysed by EDS.

Coating procedure

Substrates were coated in an RF (13.56 MHz) vacuum plasma system at different pressures. The RF vacuum plasma experimental setup is shown in Figure 1.

The sample surfaces were coated by plasma grafting of sprayed PTFE. Dimensions of the reactor are 450 mm inner diameter and 500 mm height. A 180 mm diameter and 0.8 mm thick disk-shaped steel electrode was used for the PTFE coating process by vacuum plasma. The electrode was placed parallel to the surface of the substrate. The distance between the electrode and substrate surface was set to 20 mm. Ar and He gases were used for surface activation and coating. The samples and vacuum reactor were cleaned with ethyl. Afterwards, substrates were placed under the electrode at 20 mm distance for surface activation. The system base pressure was 1×10<sup>-3</sup> Torr (0.133 Pa). The surface activation process was carried out using Ar gas atmosphere. The pressure of the system was set to 10 mTorr, then argon gas flowed in for 30 minutes to remove the oxygen and nitrogen. The activation process parameters were time of 10 min, power of 180 W, pressure of 10 mTorr, and RF frequency of 13.56 MHz. After the

activation process, the pressure of the system was set to atmospheric pressure. After opening the shutter of the reactor, PTFE material was hand sprayed onto the sample surface in an argon gas environment. Then, the shutter of the reactor was shut down and the pressure of the reactor was reduced to 1×10<sup>-3</sup> Torr. After the gas flow, the plasma was applied at different parameters. These parameters are given in Table 2.

In the second process, a thin layer was deposited by hexafluoropropene (C<sub>3</sub>F<sub>6</sub>). Two different steel electrodes were used for the C<sub>3</sub>F<sub>6</sub> coating process by vacuum plasma. The distance between electrode and substrate surface was set to 20 mm for the two electrodes. First, surface activation was carried out, as in the PTFE coating process. After the surface activation process, the pressure of the reactor was reduced to 1×10<sup>-3</sup> Torr. C<sub>3</sub>F<sub>6</sub> monomer was flowed in, and then plasma was applied at different parameters, as shown in Table 3.

Wear tests

The effect of RF vacuum plasma treatment on the parameters of the surface properties of the substrate materials as well as the effect of the coating materials on the tribological properties of PTFE and C<sub>3</sub>F<sub>6</sub> coated carbon steel were investigated. Wear tests were carried out on a tribotester under dry conditions. The process parameters for wear tests were 8 mm/s sliding speed, 1 N normal load and 3 m sliding length. The ceramic ball and surfaces of substrates were cleaned with alcohol before each test. Wear tests were carried out at a temperature of 22 ± 2°C and relative humidity of 60 ± 5%. Sample surfaces were examined under optical microscope for investigating the wear scars. After the wear tests, all sample surfaces were analysed by SEM.

Results and Discussion

PTFE and C<sub>3</sub>F<sub>6</sub> polymeric materials were coated onto heat treated AISI 1050 carbon steel. Wear tests were carried out using a tribotester. Below are the tribological properties of PTFE and C<sub>3</sub>F<sub>6</sub> coated on the carbon steel substrate.

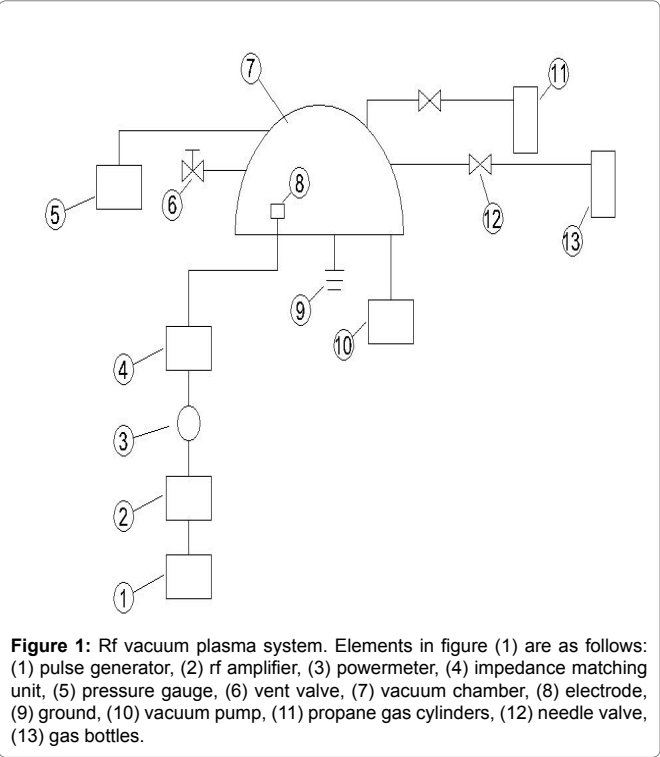


Figure 1: Rf vacuum plasma system. Elements in figure (1) are as follows: (1) pulse generator, (2) rf amplifier, (3) powermeter, (4) impedance matching unit, (5) pressure gauge, (6) vent valve, (7) vacuum chamber, (8) electrode, (9) ground, (10) vacuum pump, (11) propane gas cylinders, (12) needle valve, (13) gas bottles.

Table 1: Surface treatments applied to the substrates.

Process	Average hardness of substrate
Full softened	HRB 87
Shot peened	HRC 40
Quenched at + 210°C and tempered	HRC 59
Quenched at + 500°C and tempered	HRC 37

Table 2: Plasma parameters of the PTFE coating process.

Sample number	Power (W)	Pressure (mTorr)	Gas type	Operation time (min)
(a)	180	100	Ar	7.5
(b)	180	50	Ar	7.5
(c)	100	100	Ar	7.5
(d)	100	100	He	7.5
(e)	100	100	Ar	15
(f)	180	50	Ar	15

Table 3: Plasma parameters of coating process for C<sub>3</sub>F<sub>6</sub>.

Sample number	Power (W)	Pressure (mTorr)	Operation time (min)
(a)	50	300	15
(b)	100	300	15
(c)	50	100	15
(d)	50	300	30
(e)	100	100	30

## Optical images

Cross-sections of coated substrates were investigated. The coating thicknesses of coated substrates were measured as average values. Coating thicknesses of PTFE coated substrates are given in Figure 2 and coating thicknesses of  $C_3F_6$  substrates are given in Figure 3.

Argon gas was used in the PTFE coating process and gaseous coating material  $C_3F_6$  plasma was used during the  $C_3F_6$  coating process. When the power was decreased, the coating layer thickness decreased, as shown in Figures 2 and 3. The vacuum pressure is another process parameter that affects the coating thickness. When the pressure was decreased, the coating thickness also decreased on PTFE coated substrates. In general, for each coating material (PTFE and  $C_3F_6$ ) the thickness of the coating increases as time increases.

## EDS analysis of substrates

EDS analysis of coated and non-coated substrates was carried out. EDS analysis results for the non-coated substrates are shown in Figure 4. The AISI 1050 carbon steel sample is composed of Fe and C. The red colour represents Fe.

EDS analysis results for PTFE coated substrates are shown in Figure 5. The coating material contains flourine (F) and carbon (C)

elements. Figure 5 shows that the value of F/C ratio decreases when coating power decreases. Plasma coating density increases as the processing time is increased. During the coating process, when the power and processing time were kept constant and process pressure was decreased, the F/C ratio decreased. The figure also shows the distribution of atoms on the sample surface. When the processing time and pressure were kept constant and the power increased, the F/C ratio decreased. In addition, when power and pressure were kept constant and processing time was increased, the F/C ratio decreased. The distribution of C and F atoms was homogeneous. This is due to homogeneous coating and suitable substrate location.

## Wear tests of PTFE and $C_3F_6$ coatings

Substrates were coated with PTFE and  $C_3F_6$  using a vacuum plasma coating method. After the coating process, tribological properties of samples were investigated by using a tribotester. Friction coefficient values were obtained by wear tests. Little change occurred when power, pressure and treatment time were changed, as shown below in Figure 6. The measurements were carried out for different substrate surface hardness conditions. Fully softened, quenched at +210°C and tempered, and quenched at +500°C and tempered substrate hardness conditions all have close friction coefficients. This is explained by

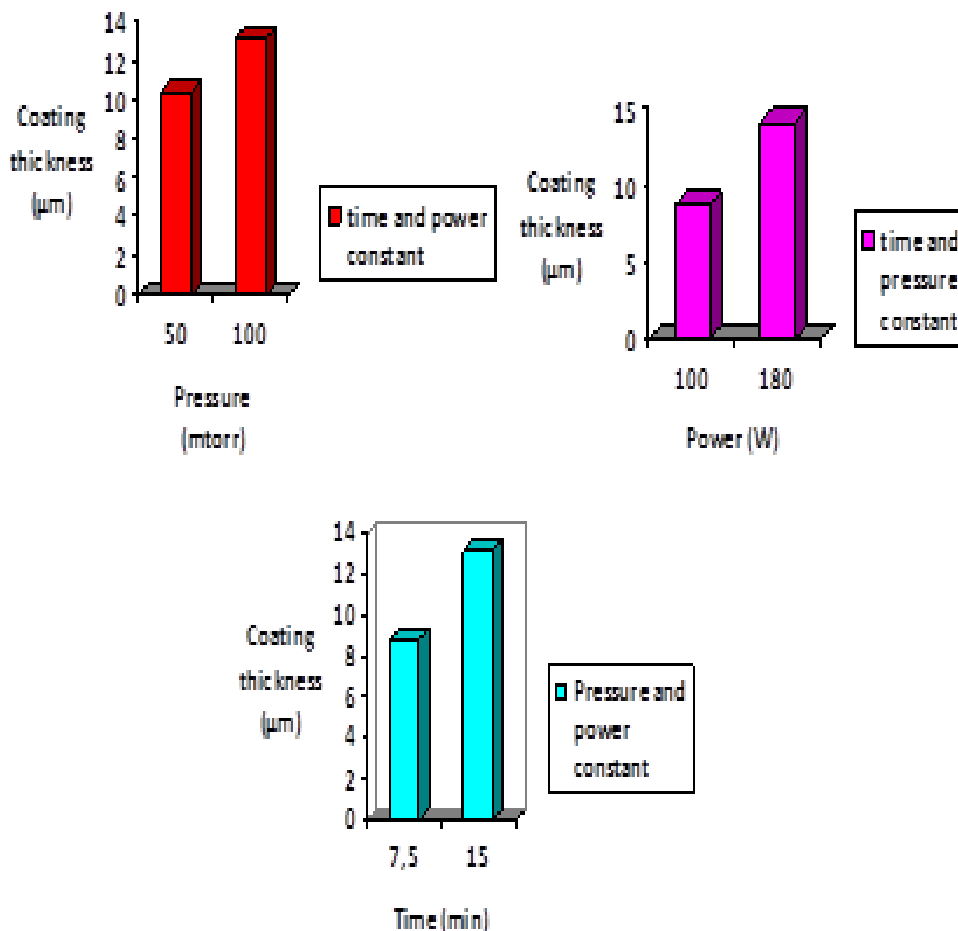


Figure 2: Coating thickness of PTFE coated substrates.

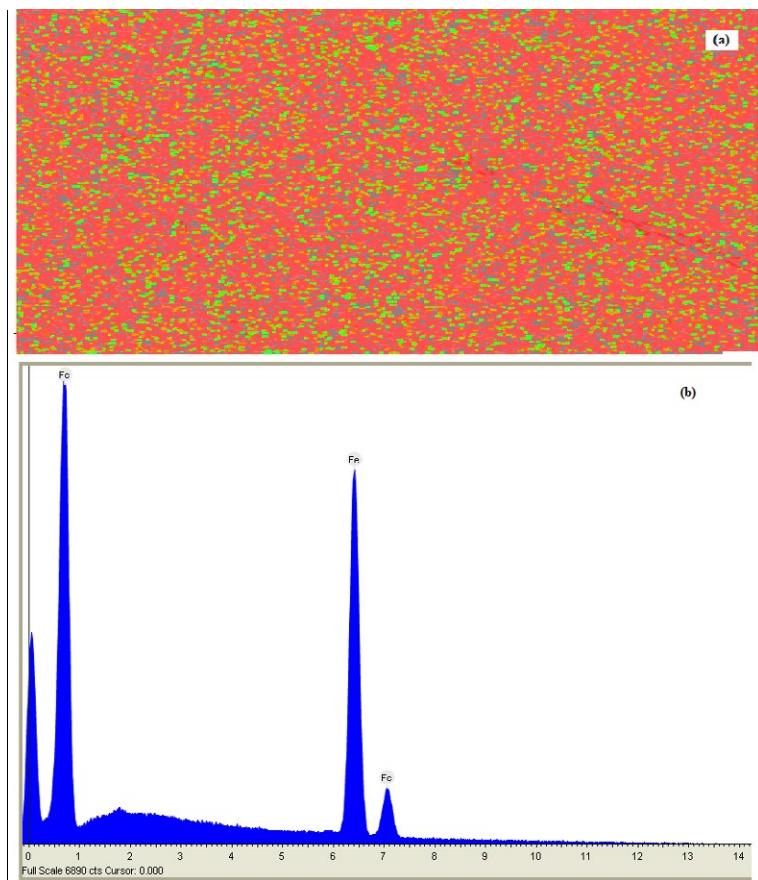


Figure 3: EDS analysis of non-coated substrates.

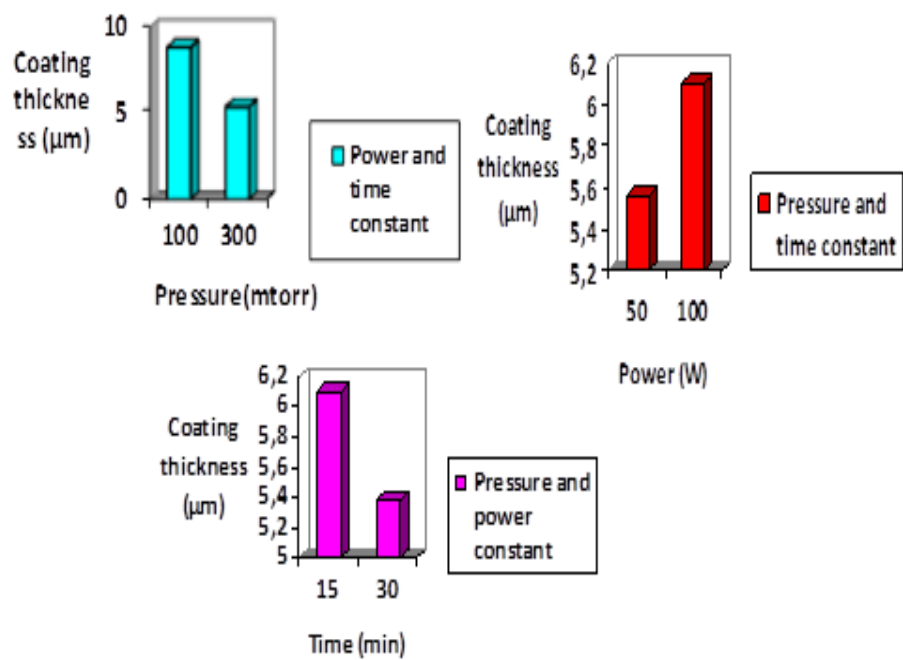


Figure 4: Coating thickness of C<sub>3</sub>F<sub>6</sub> coated substrates.

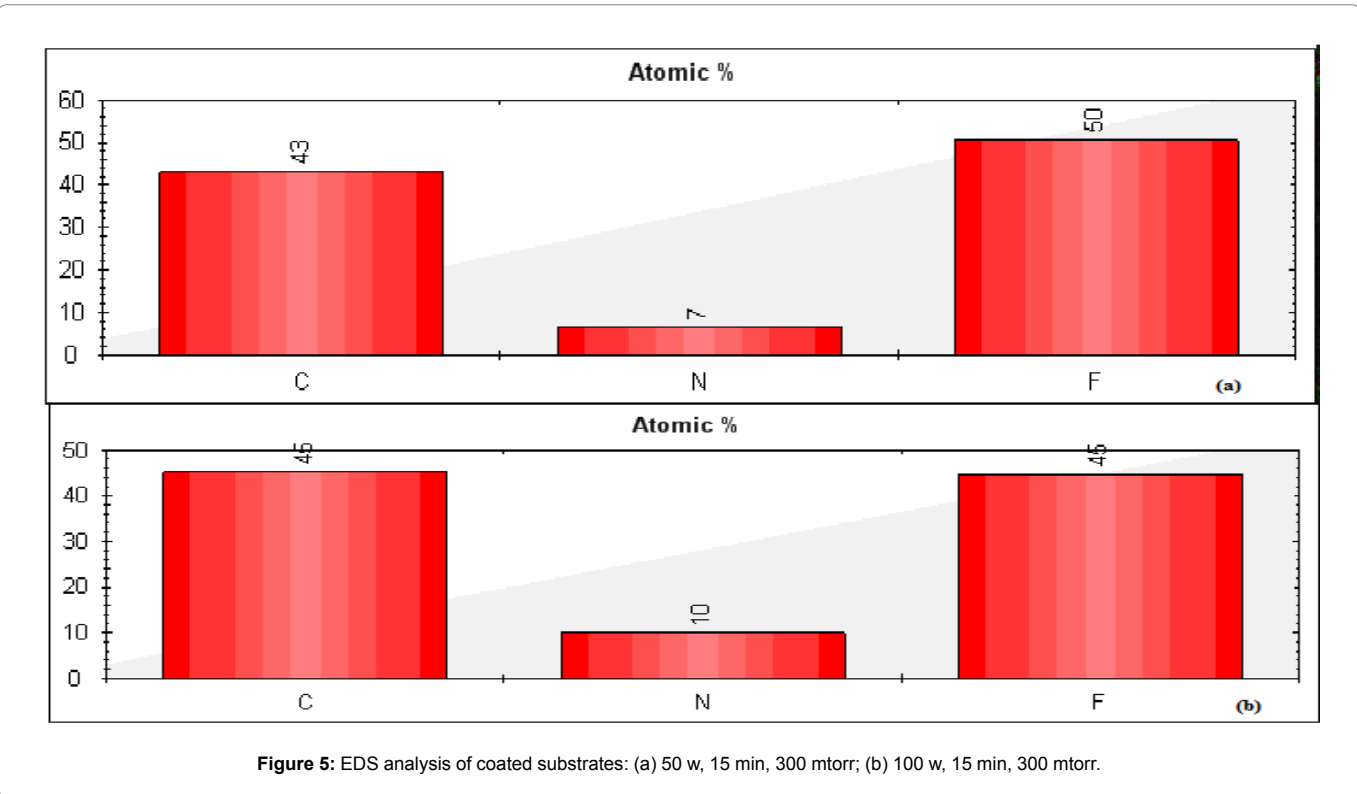


Figure 5: EDS analysis of coated substrates: (a) 50 w, 15 min, 300 mtorr; (b) 100 w, 15 min, 300 mtorr.

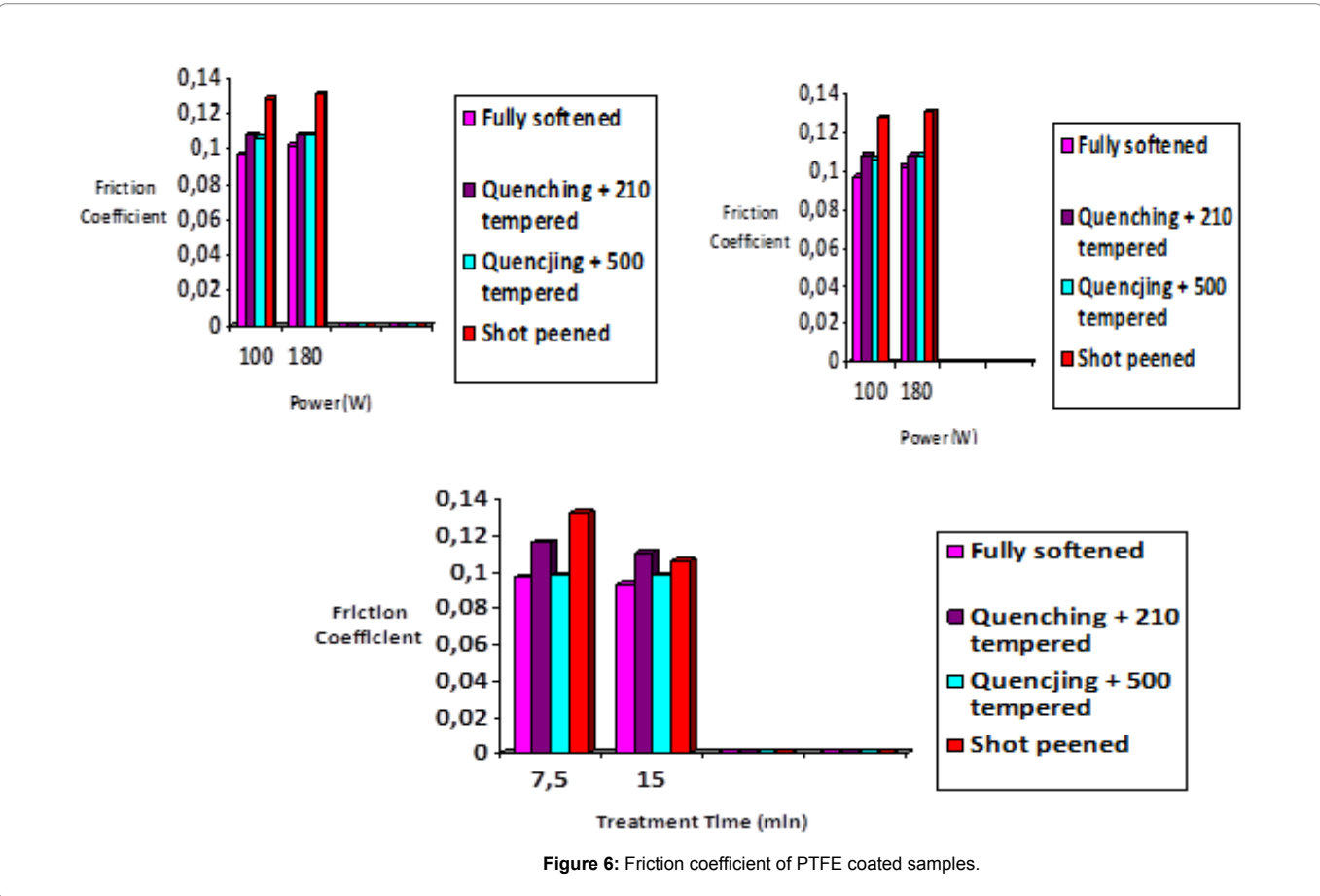


Figure 6: Friction coefficient of PTFE coated samples.

the linear elastic behaviour of the coating and substrate material. However, shot peened substrates have higher friction coefficients than the other substrates for different process parameters.

Changes in friction coefficients of  $C_3F_6$  coated substrates are given below in Figure 7. Friction coefficients of all substrates increased with increasing discharge power. When pressure was increased from 100 mTorr to 300 mTorr, friction coefficients of the substrate quenched at +500°C and tempered and the fully softened substrates were increased, and friction coefficients of the substrates quenched at +210°C and tempered were decreased. Friction coefficient values for shot peened substrate were lower than for the other substrates for all process parameters.

Also in this study the friction force values were obtained with a tribotester. In general, friction coefficient and friction force values are higher at the beginning of wear experiments but after reaching the regime friction force the values remain constant during the experimental period. In general, friction force and friction coefficient are higher for  $C_3F_6$  coated substrates than for PTFE coated substrates.

### Optical microscope examination of wear traces

Wear traces of substrates which were coated with PTFE and  $C_3F_6$  under vacuum conditions were investigated using an optical microscope. Average wear trace widths of PTFE coated substrates are given in Figure 8.

Wear traces of fully softened substrates coated with PTFE at different plasma parameters are given in Figure 9a. The coating shield acts like a lubricant that prevents ceramic ball deformation and ball-metal contact. Wear traces of the substrates quenched at +210°C and tempered, coated with PTFE at different plasma parameters, are given in Figure 9b. Wear trace widths of samples were measured, with the wear trace patterns more obvious, uniform and homogeneous. Furthermore, wear trace pattern widths were smaller than for fully softened samples. Wear traces of PTFE coated samples quenched at +500°C and tempered are given in Figure 9c. Figure 9c shows the wear traces of samples quenched at +500°C and tempered, which are similar those of the samples quenched at +210°C and tempered. In addition, wear trace widths were narrower and wear traces were more obvious than in the fully softened case. Wear traces of PTFE coated shot peened samples are given in Figure 9d. Bright areas on shot peened samples are due to substrate-ceramic ball contact.

In addition, measurements of wear traces of PTFE coated samples at different hardnesses at constant process parameters (180 W, 100 mTorr pressure Ar for 7.5 min) were taken. The influence of surface quality and surface hardness of substrate on surface coating quality was determined. Wear traces of all samples are given in Figure 10. The traces of fully softened and shot peened substrates show bright regions. This is explained by inadequate coating quality. Coating

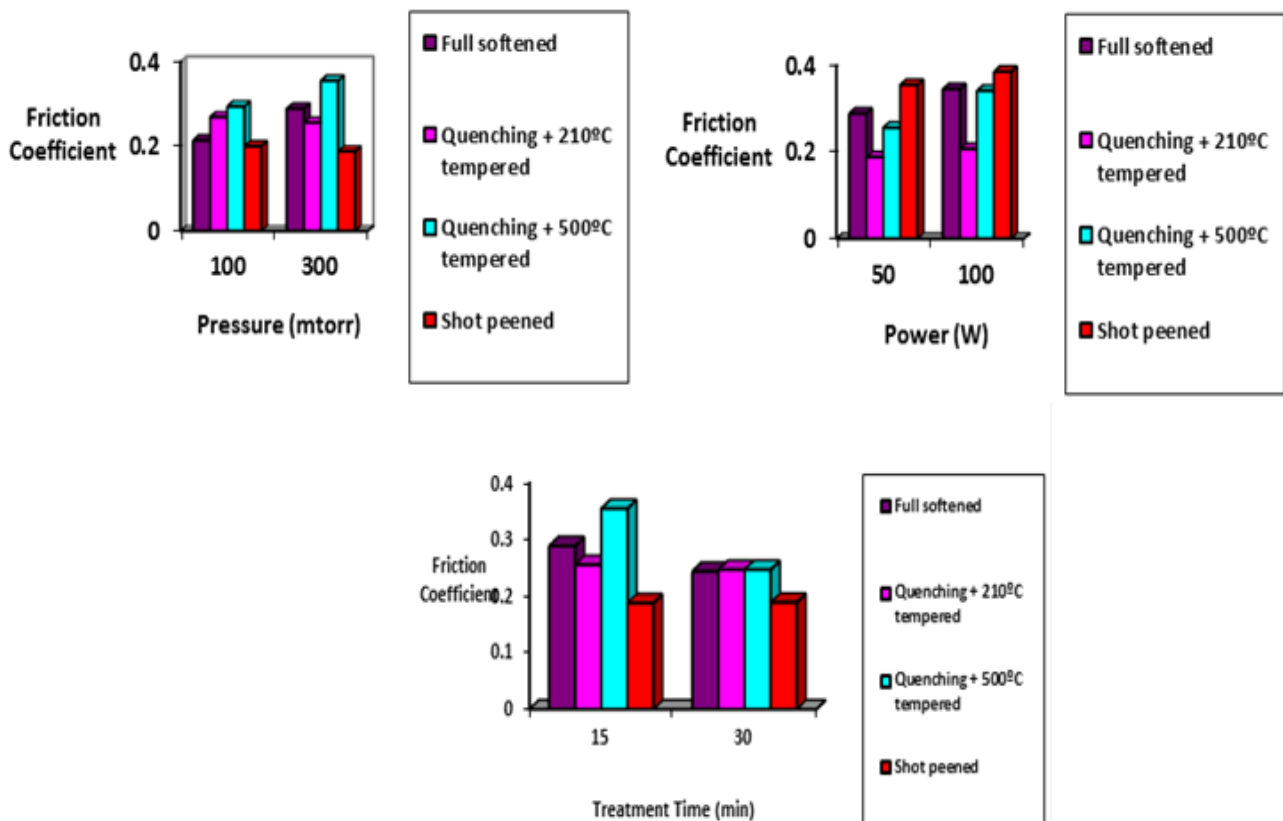


Figure 7: Friction coefficient of  $C_3F_6$  coated samples.



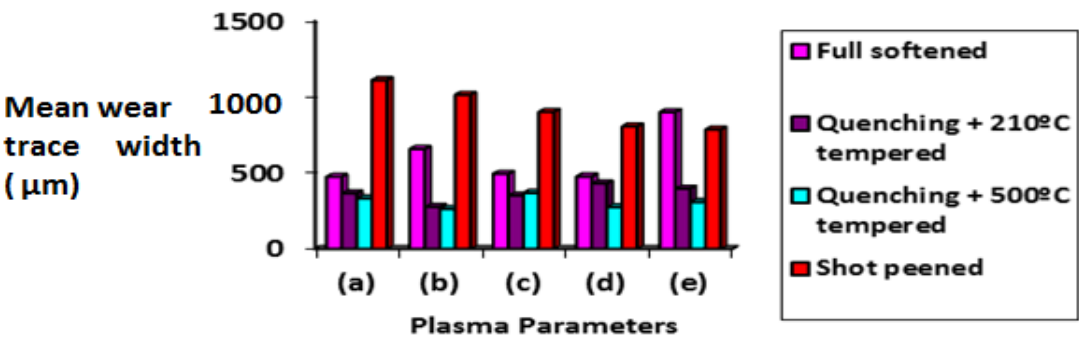


Figure 8: Average wear trace widths of ptfе coated substrates: (a) 180 w, 100 mtorr pressure ar for 7.5 min; (b) 180 w, 50 mtorr pressure ar for 7.5 min; (c) 100 w, 100 mtorr pressure ar for 7.5 min; (d) 100 w, 100 mtorr pressure he for 7.5 min; (e) 100 w, 100 mtorr pressure he for 7.5 min.

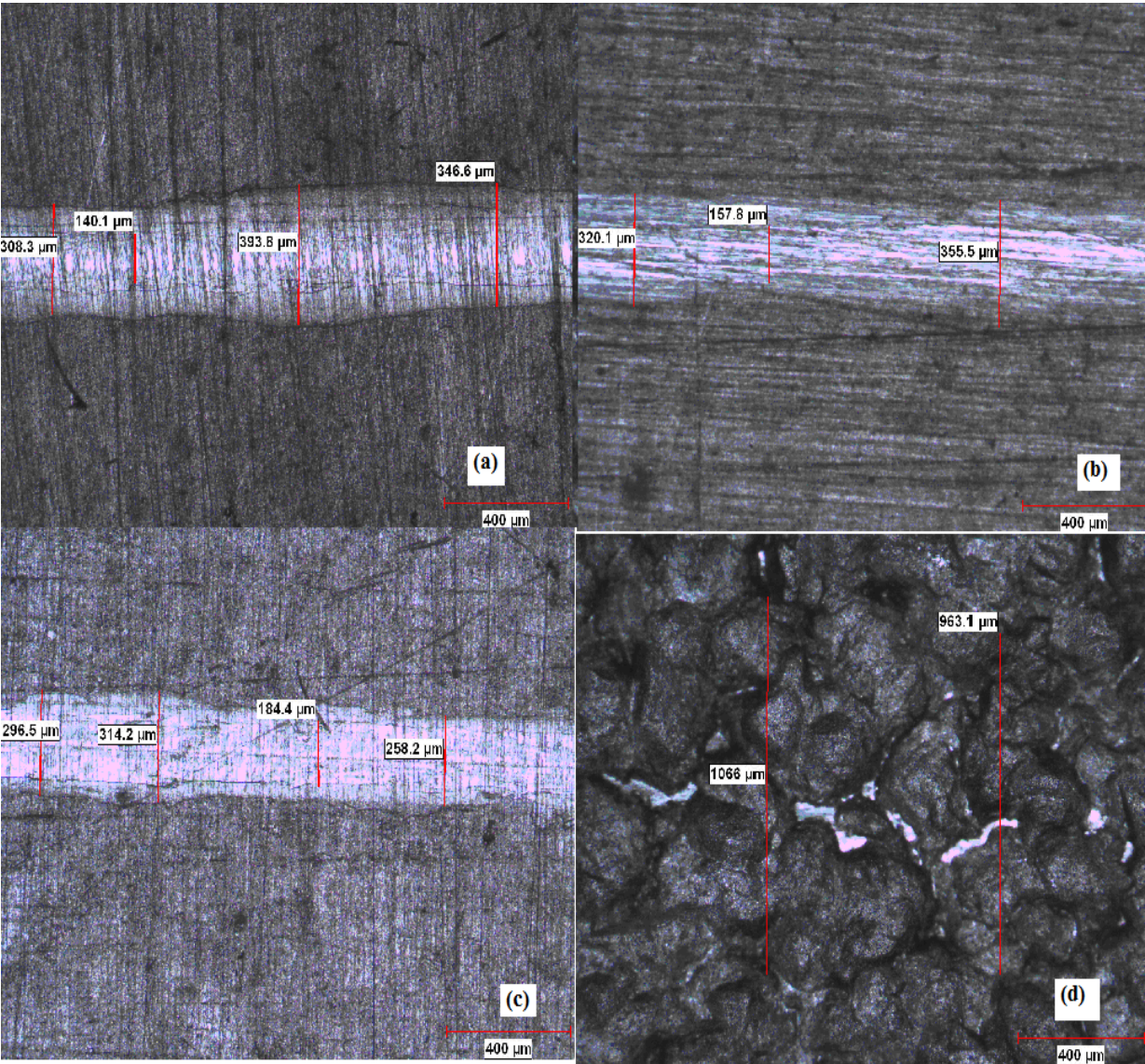
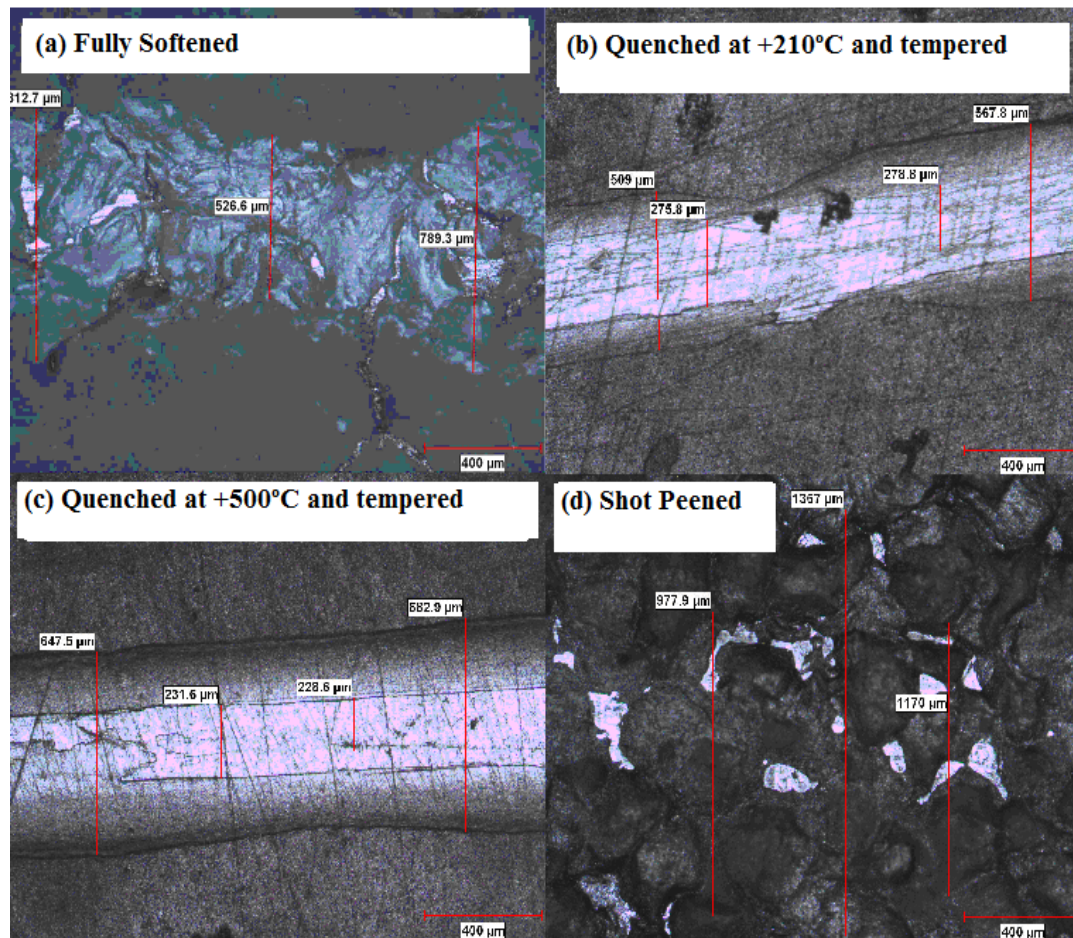


Figure 9: Wear traces of PTFE coated samples: (a) fully softened; (b) quenched at +210°C and tempered; (c) quenched at +500°C and tempered; (d) shot peened.





**Figure 10:** Wear traces of all samples at constant plasma parameters (180 w, 100 mtorr ar, 7.5 min): (a) fully softened; (b) quenched at +210°C and tempered; (c) quenched at +500°C and tempered; (d) shot peened.

material wears immediately followed by indication of metal-ceramic friction. Moreover, wear trace widths for fully softened and shot peened substrates were found to be larger than for other substrates.

$C_3F_6$  coating material was compared to PTFE coating material. Wear traces of fully softened and shot peened substrates are given in Figure 11a-11d. Coating material piled up at the edge of the wear trace in fully softened substrates. This can be explained by the inadequate coating quality. For shot peened substrates, surface roughness causes peak areas to be worn out first. This was seen in all shot peened substrates. To prevent this problem, a very thin layer of substrate can be machined by grinding before treatment. Wear traces of substrates quenched at +210°C and tempered and quenched at +500°C and tempered are given in Figures 11b and 11c. Bright regions are narrower than in the case of fully softened substrates. In addition, substrates quenched at +210°C and tempered were coated with  $C_3F_6$  at different process pressures. Metal-ceramic contact did not occur at 300 mTorr pressure. Wear was reduced by increasing the treatment pressure. Wear traces of substrates quenched at + 500°C and tempered also show reduced wear at higher pressure.

Samples with different hardness values were coated with  $C_3F_6$  at 50 W, 15 min, 300 mTorr plasma process parameters. More bright regions were observed in shot peened substrates. This is explained by

the fact that shot peened substrate cannot be coated easily compared to the other substrates, as can be seen in Figure 12.

Also in this study fully softened (a) and shot peened (b) substrates were coated with PTFE and  $C_3F_6$  at 100 W, 15 min, 100 mTorr plasma parameters (Figures 13a and 13b). Quenched at +210°C and tempered (a) and quenched at +500°C and tempered (b) substrates were coated with PTFE and  $C_3F_6$  at the same plasma parameters (100 W, 15 min, 100 mTorr) (Figures 13a and 13b).

Wear trace widths of PTFE polymer materials are smaller than the wear trace widths of  $C_3F_6$  polymer materials. This is independent of surface quality and the hardness values of coating materials. Wear trace size of PTFE coated samples reveals the most appropriate substrate by comparison between all PTFE coated samples. PTFE coated substrates quenched at +210°C and tempered have smaller wear trace width. Surface roughness of substrates quenched at +210°C and tempered is the same as for substrates quenched at +500°C, tempered and fully softened. It is concluded that surface roughness values of the samples do not affect the coating quality. When the hardness values of the substrate were increased, wear trace widths were found to decrease. This is due to smaller elastic deformation. In addition, the coating had a low friction coefficient. Larger trace widths indicate higher coefficient of friction values.



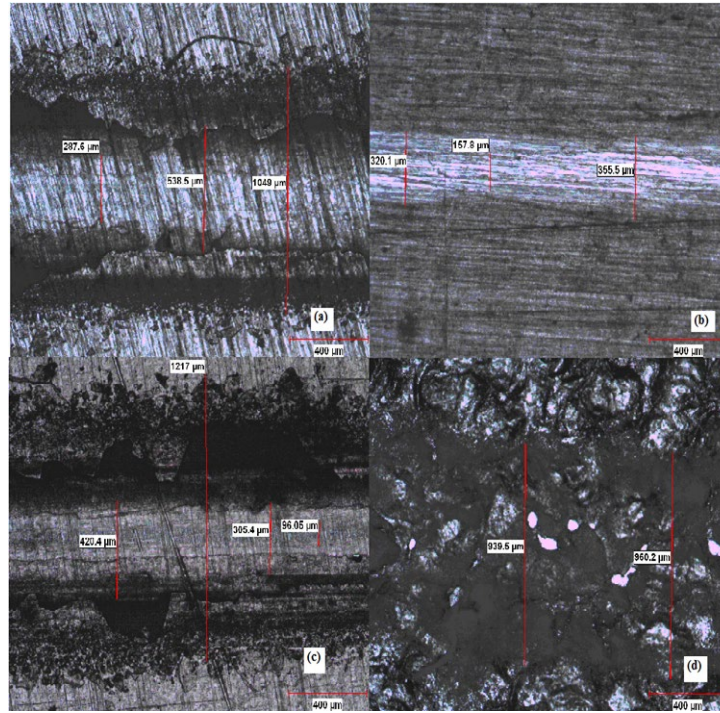


Figure 11: Wear traces of  $C_3F_6$  coated samples: (a) fully softened; (b) quenched at  $+210^\circ\text{C}$  and tempered; (c) quenched at  $+500^\circ\text{C}$  and tempered; (d) shot peened.

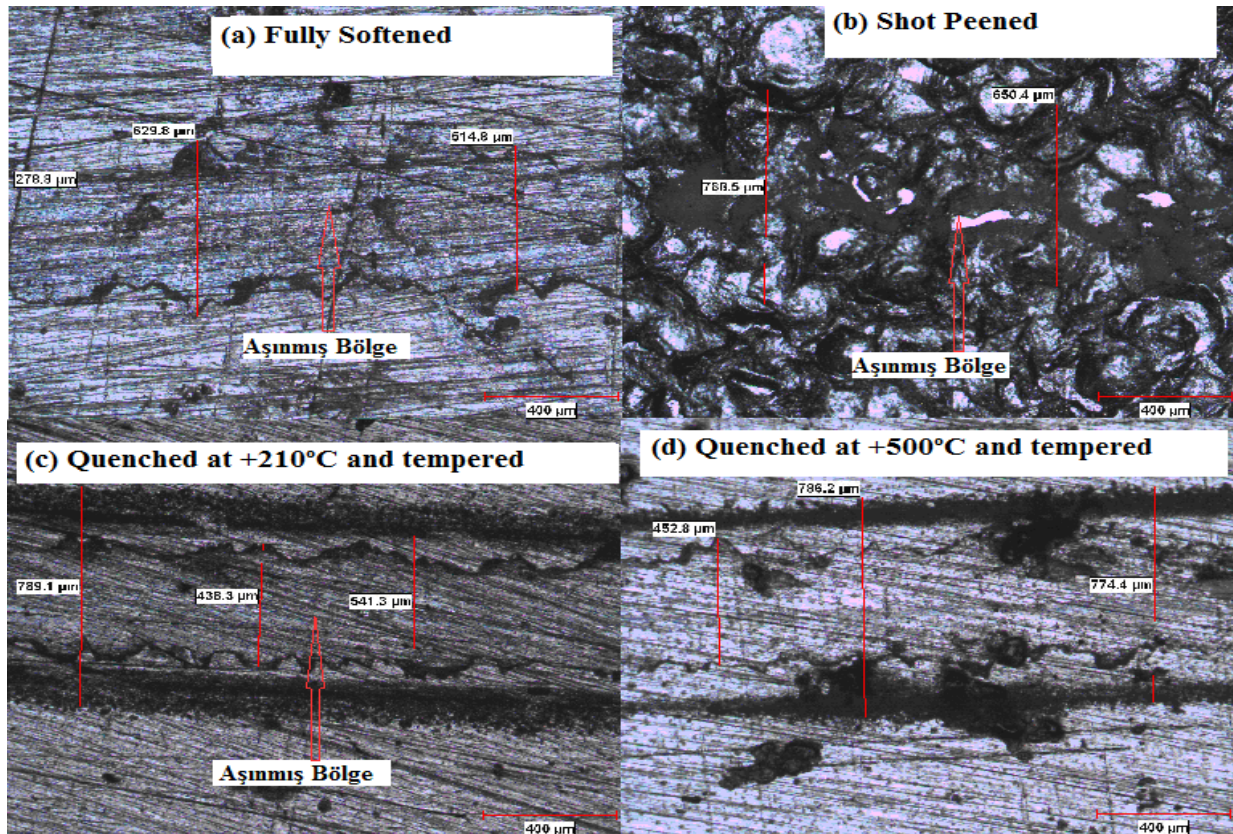
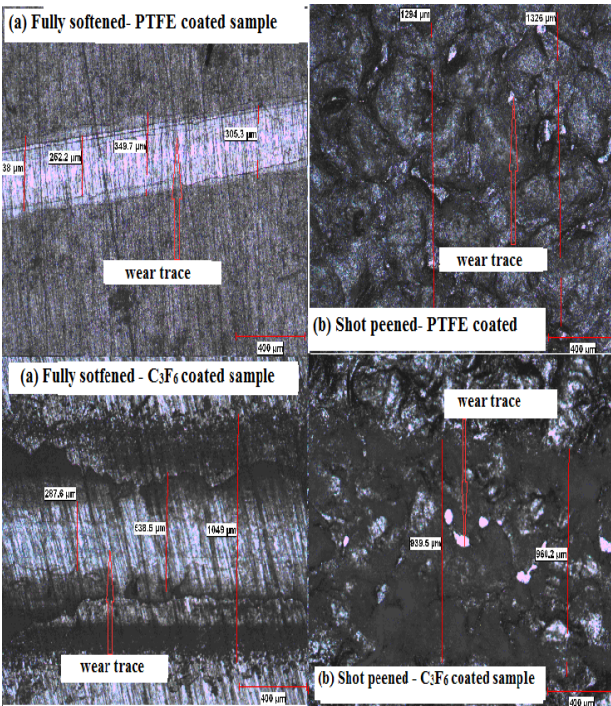
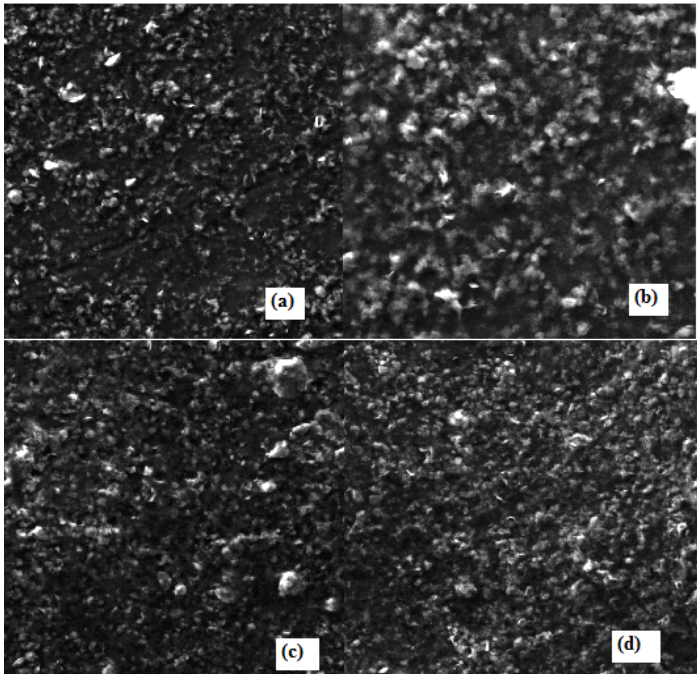


Figure 12: Wear traces of all samples at constant plasma parameters (50 w, 15 min, 300 mtorr): (a) fully softened; (b) shot peened; (c) quenched at  $+210^\circ\text{C}$  and tempered; (d) quenched at  $+500^\circ\text{C}$  and tempered.





**Figure 13:** Wear traces of PTFE and C<sub>3</sub>F<sub>6</sub> coated samples (100 w, 15 min, 100 mtorr): (a) fully softened substrate (PTFE and C<sub>3</sub>F<sub>6</sub> coated) and (b) shot peened substrate (ptfe and C<sub>3</sub>F<sub>6</sub> coated).

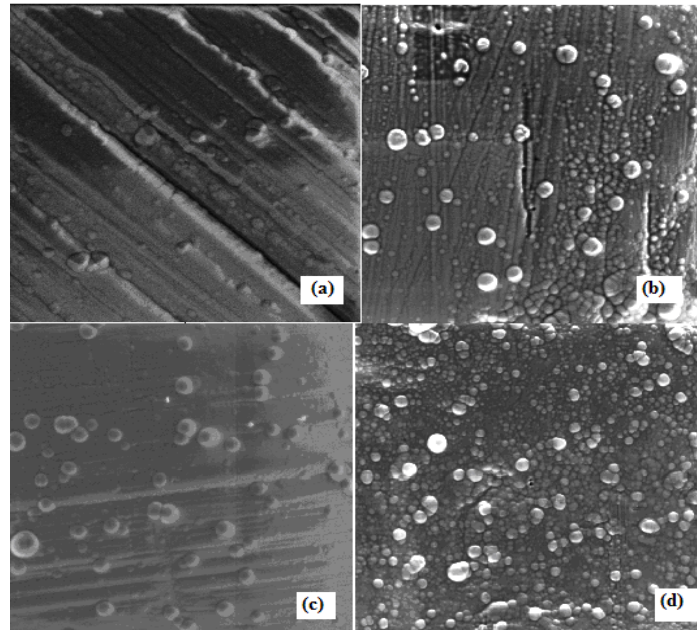


**Figure 14:** Sem images of ptfе coated samples: (a) fully softened; (b) quenched at +210°C and tempered; (c) quenched at +500°C and tempered; (d) shot peened.

**SEM examination of wear traces**

Two different coating materials (PTFE and C<sub>3</sub>F<sub>6</sub>) were coated onto the same kinds of substrates at vacuum conditions. The coating materials have different characteristics. This difference is seen clearly

in SEM images of PTFE and C<sub>3</sub>F<sub>6</sub> polymer materials. SEM images of PTFE coated samples show that wear regions have homogeneous structure with larger wear debris seen in coatings with fine grains (Figure 14).



**Figure 15:** Sem images of  $C_3F_6$  coated samples: (a) fully softened; (b) quenched at +500°C and tempered; (c) quenched at +500°C and tempered; (d) shot peened.

Images of  $C_3F_6$  coated samples are given in Figure 15. There are small loose spherical particles, as shown in the figure. The presence of these particles increases the friction coefficient value.

## Conclusion

Heat treated and shot peened AISI 1050 carbon steel substrates were coated with PTFE and  $C_3F_6$  polymer materials at different plasma parameters in an RF vacuum plasma system. After the coating process, wear properties of the samples were investigated. Based on the results obtained from this study, the following conclusions may be made [12]:

- (1) The friction coefficient value of PTFE coated fully softened substrates is approximately 4.2 times lower than that of  $C_3F_6$  coated substrates. The friction coefficient values of PTFE coated substrates quenched at +210°C and tempered, quenched at +500°C and tempered, and shot peened were 3.5, 3, and 2 times lower, respectively, than those for  $C_3F_6$  coated substrates. Therefore, PTFE polymer material is superior to  $C_3F_6$  polymer material for coating.
- (2) The surface quality of the substrate affects the coating quality. Shot peened substrates have a rough surface; therefore, the coating quality of these substrates is low. The coating quality of shot peened samples can be increased by grinding or machining after the shot peening process. In this study, samples quenched at +210°C and tempered were the best substrates for the coating process.
- (3) Plasma process parameters do not affect the coating quality.

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