

Atmospheric Pressure Plasma Treatment of Amorphous Polyethylene Terephthalate for Enhanced Heatsealing Properties

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Abstract

An atmospheric pressure plasma system has been used to treat amorphous polyethylene terephthalate (APET) to enhance its heatseal properties to a polyethylene terephthalate (PET) film. The plasma treated APET sheet material was thermoformed into trays for use in the food packaging industry and heatsealed to a PET film. The heatsealing properties of the resulting package were assessed using the burst test technique. It was found that the plasma treatment significantly enhanced the adhesive properties and an increase in burst pressure from 18 to 35 kPa was observed for plasma treated food trays. The APET surface chemistry was assessed after plasma treatment where it was found that the plasma treatment had affected an increase in oxygen and an addition of nitrogen species to the polymer surface. The surface roughness (R_a) of the plasma treated samples was also observed to increase from 0.4 to 0.9 nm after plasma treatment.

Introduction

Heatsealed polymer trays are widely used for packaging food products, particularly meat and fish [1]. In the heat sealing process a polymer top film is applied to the polymer tray under elevated temperature and pressure in order to enclose the food product in an airtight environment [2]. The conventional method for creating a strong bond between a food tray and a top film is to use a co-extruded heatseal layer (Figure 1).

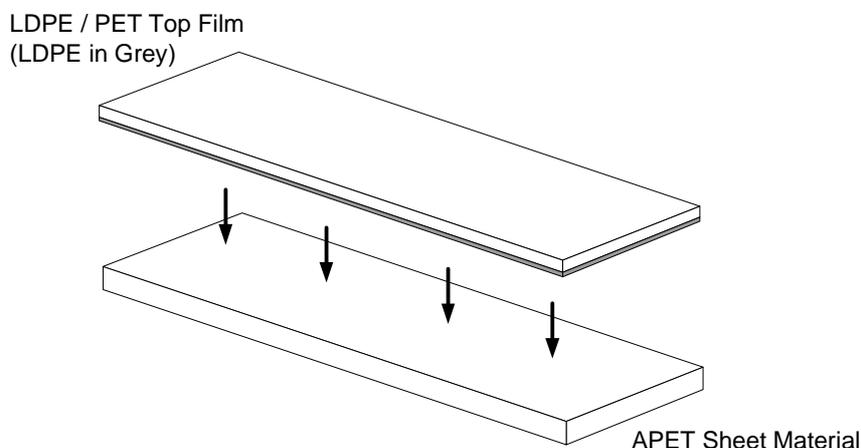


Figure 1: Conventional heatseal technique where a low density polyethylene (LDPE) layer of lower melting temperature is utilised to fuse the two PET polymers together.

This layer has a lower melting temperature than that of the top film and thus during the heatsealing operation when this layer is brought into contact with the food tray it is melted, thereby sealing the top film to the food tray [2]. An example of this heatseal layer is low density polyethylene (LDPE), which is co-extruded with the polyethylene terephthalate (PET). The melting temperatures of LDPE and PET are 120 and 250°C respectively. The PE layer due to its lower melting temperature (T_m) substantially enhances the adhesion between this top film and PET tray materials. This co-extruded film however cannot be separated after product use, which means the packaging cannot be recycled [3, 4].

The focus of this paper is to investigate if plasma surface modification of the polymer surface can be used as a technique for enhancing heatseal bond strength in the absence of a lower melting point heat seal layer. The plasma treatment approach was investigated as plasmas have been widely applied to enhance the surface energy of polymers particularly prior to adhesive bonding [5, 6]. The depth of modification for plasma treatment is generally less than 10 nm [7]. One of its effects is to remove weak boundary layers hence strengthening the adhesive bond [8]. Chain scissioning of the long polymer molecules may also occur, thus reducing the level of polymer crystallinity and generating chemical sites which are available for bonding with an adhesive. For example the incorporation of functional groups containing oxygen and nitrogen into the surface has been demonstrated after plasma activation [9]. It has been shown that even if only a few chemical sites are created there will be a large increase in adhesive strength [10]. Removal of surface contaminants is also an important contribution of plasma treatment to polymer adhesion [11]. While there have been no previous reports on the use of plasma treatments as a means of enhancing heatseal bond strength in food packaging, a study by Wu et al. has been carried out on a PET microchip substrates [12]. In this study it was successfully demonstrated that oxygen microwave plasma could be used to enhance the thermal bond strength between two PET layers (in the absence of an adhesive layer).

MATERIALS AND METHODS

Substrates

The polymer materials utilised in this work were amorphous polyethylene terephthalate (APET) for the tray material and polyethylene terephthalate (PET) top film. The APET material of gauge 350 μm was manufactured by Holfeld Plastics, Ireland. Conventional APET food trays are sealed to a top film of PET using a co-extruded low density polyethylene (LDPE) layer. The PET film investigated in this work is a mono layer PET film of gauge 50 μm supplied by Versatile Packaging, Ireland. In this study initial trials were carried out using rectangular strips of APET (25 x 75 mm) and PET top film (25 x 125 mm). Subsequent trials were carried out with APET food trays with dimensions 194 x 145 x 38 mm, bonded to 250 cm wide PET sheet (50 μm thick).

Plasma Treatments

The plasma treatments were carried out using three Dow Corning atmospheric pressure plasma systems; LabLine™, AP4™ and PlasmaStream™.

The LabLine is a pilot-plant system which can treat materials up to 300 mm wide as described previously [13]. The LabLine electrodes comprise of an outer frame

of epoxy/laminate structure (Micam©) and a toughened glass dielectric. The conductive component is a salt water solution. The electrodes measure 320 x 300 mm with an electrode gap of 5 mm. The plasma is powered by a low frequency generator (approx. 20 kHz) which delivers up to 2000 W.

The Dow Corning AP4 system is an industrial scale atmospheric pressure plasma processing tool [14]. The system consists of two electrode pairs, each pair generating 3 plasma zones, with each zone measuring 225 x 1200 mm. The electrodes are powered up to 6500 W in parallel with the inner electrodes 180° out of phase with the outer electrodes. The electrodes consist of a conductive liquid housed within a glass / alumina / epoxy composite dielectric perimeter with an electrode gap of 5 mm. The input parameters to the plasma such as applied power, gas flow rate and polymer through-put speed are controlled using a programmable logic controller (PLC) system. This allows for rapid set up times and minimises down time between treating rolls of polymer. Both reel to reel plasma systems are shown in Figure 2.

The atmospheric pressure PlasmaStream has a delivery applicator consisting of a 75 mm long by 15 mm wide Teflon tube [15, 16]. The systems pin electrodes are connected to a high voltage single phase inverter generator (maximum 15 kV), operating between 15 - 25 kHz that creates rectangular pulses. Manual valved rotameters are used to control the He flow rate (0 – 30 L/min) through the nozzle. The plasma treatment can be applied over an area of approximately 200 x 200 mm and the Teflon nozzle to substrate distance can be controlled by positioning the head using a CNC controller.



Figure 2: LabLine & AP4 reel-to-reel atmospheric pressure plasma systems for the treatment of webs up to 0.3 and 1.2 Meters wide respectively

Characterisation

The changes at the surface of the plasma treated polymer surface were monitored using the surface energy technique, atomic force microscopy (AFM) and x-ray photoelectron spectroscopy (XPS).

A Dataphysics OCA 20 system was used to analyse the surface energy of both untreated and plasma treated polymers. This system facilitates the measurement of the contact angle of liquids dropped onto the sample surface. The contact angle is measured by placing the droplet on the surface of the polymer using the integrated dispenser system; the angle which this drop makes with the surface is then measured using the associated high resolution camera and computer software. The liquid drop properties are extracted using the Strom et al. [17, 18] technique. In order to determine the surface energy it is necessary to dispense three liquids of different polarities. In this study deionised water, ethylene glycol and diiodomethane were

used. The measured contact angles are then input into the Owens, Wendt, Rabel and Kaelble (OWRK) software model [19] and the surface energy value in mN/m is obtained. Each measurement was carried out in triplicate in order to minimise error and analyse the treatment over a wider surface area.

A CP-II (Veeco) atomic force microscope (AFM) was operated with a 100 μm scanner and scan rate of 2 Hz. The AFM images were recorded in non-contact mode with silicon (phosphorous doped) cantilevers (nominal spring constant = 40 N/m, tip radius <10 nm).

XPS analysis was performed using an Axis Ultra spectrometer system (Kratos Analytical). Samples were irradiated with monochromated x-rays (Al Ka, 1486.6 eV) and photoelectrons were analysed from a selected area 700 by 300 μm , with a take-off-angle of 90°. To obtain good spectra the instrument's charge neutralization system was used. The magnetic field generated by the systems magnetic immersion lens is used to confine low energy thermionically emitted electrons from a filament and transport them to the sample surface. Each analysis position was analysed in the survey mode (Pass Energy 160 eV) to determine the elements that were present at the surface and their relative concentrations. CasaXPS (Casa Software Ltd) data processing software was used to calculate the area under peaks representative of elements detected, these were then normalized to take into account relative sensitivity and to provide relative concentrations. Each analysis position was also analysed in the high resolution mode (Pass Energy 20 eV) to extract more detailed information on the elements present at the surface. Charge correction was conducted using a method of internal standard. Peaks representative of known chemical states were fitted to high resolution core level spectra. The measured binding energy of these peaks (BE_{meas}) was used with a relevant reference binding energy (BE_{Ref}) by the CasaXPS (Casa Software Ltd) data processing software to apply correction energy, E_{corr} to all the spectra acquired from the same analysis position.

Thermoforming

The thermoforming of food trays from the plasma treated PET sheet material was carried out using plug assisted vacuum Illig and TFT thermoforming units. Standard conditions for thermoforming APET were used. The temperature on the polymer surface was measured using heat sensitive strips and was found to be approx 90°C.. At this temperature the amorphous portion of the PET polymer becomes more flexible, which facilitates the forming process (Glass Transition Temp, $T_g = 75^\circ\text{C}$)

Heatseal

The plasma treated PET material was heatsealed to the PET film using two systems. The HSE3 sealer was used to heatseal flat PET samples which were plasma treated but not thermoformed. The Orved sealer was used to apply films to food trays which were thermoformed from plasma treated PET material.

Testing of rectangular polymer samples measuring 100 x 25 mm was carried out on the RDM HSE3 heatsealer apparatus. For these peel test samples an area of 25 x 25 mm was bonded. The HSE3 is a dedicated heatsealing machine with full control over the pressure, temperature and dwell time of the bond.

The Orved tray heatsealer is a manually operated heated plate which is lowered onto a top film and a food tray such that the film is bonded to the top of the tray. Interchangeable aluminium dies are used such that films can be applied to different size trays. The temperature is controlled digitally while the pressure and dwell time of this sealing system are manually controlled.

Adhesion Testing

The integrity of the increased heatseal bond strength observed after plasma treatment was assessed using both the peel test method on flat samples and the burst test technique on thermoformed food trays.

A Tinius Olsen 50 kN twin screw materials test rig was used to peel test the flat samples. Samples were mounted vertically into the tensile test apparatus with the PET film section lapping back over the APET section such that a 180° test was obtained [20, 21]. The sample peel speed was set at 30 mm/min over a peel length of 15 mm. The force required to peel these samples was divided by the width (25 mm) to obtain the peel strength [N/mm]. For samples that fully peeled with no failure of either substrate, the peel strength between 2 and 12 mm was averaged to give the value of peel strength for that heatsealed polymer. For samples that exhibited failure within the substrates the maximum peel strength observed was used, for these samples this failure always occurred in the PET top film.

The industry standard for testing the quality of this heatsealed food trays is the burst test [22, 23]. In a burst test a tray is placed into a chamber, a partial vacuum is then applied to this chamber causing the tray to expand and burst. The pressure during testing is monitored and the pressure at which the tray fails at is noted as the burst pressure. The chamber used for testing contains water and the sealing surface of the tray is submerged below this level allowing the trays to be tested in an aqueous environment. This is generally observed to degrade an adhesive bond [24]. The burst test also takes into account any areas of poor sealing brought about by the heatsealing technique as the tray will fail at the weakest point along the sealing surface.

Plasma Treatment

Initial APET heatseal studies were carried out using the LabLine system. Samples measuring 25 x 75 mm were cut from clear APET polymer sheet and were mounted on to the PET handler web using double sided tape and passed through the LabLine system. The samples were passed through the plasma chamber a number of times to increase the exposure time, the total treatment time being between 8 and 23 seconds. These flat samples were then heatsealed to a PET film using the HSE3 heatsealer and peel tested to assess their bond strength. Preliminary trials indicated that material which was plasma treated using the LabLine system exhibited enhanced adhesion to a PET top film. This enhanced sealing was observed for heatsealing temperatures above 120°C. Therefore further testing was carried out to fully assess this enhanced sealability between 120 and 190°C. Above 190°C the APET material was observed to degrade. The plasma treatment conditions initially tested to assess the bond strength of plasma treated APET were chosen based on standard operating conditions used previously on the LabLine system. These standard operating conditions are applied power of 1000W, helium flow rate of 10L/min, Oxygen flow rate of 0.2L/min and a 125 seconds treatment time.

RESULTS AND DISCUSSION

The initial plasma treatment studies were carried out on rectangular strips of APET which were heatsealed to untreated PET top film. These plasma treatment of the APET material was carried out using both the pilot plant (LabLine) and industrial scale (AP4) systems.

Pilot scale plasma treatment of APET

In order to assess the heatsealability of the plasma treated APET, samples were sealed at 7°C intervals between 120°C and 190°C using the HSE 3 heatsealer. A dwell time of 1 second was used with a pressure of 138 kPa being applied to the sealing bar. The tests were carried out using treated APET which was heatsealed to PET film with no LDPE heatseal layer present. This peel strength was tested using the 180° test method and the results are shown in Figure 3. Maximum or full adhesion for the purpose of these tests is described as the point at which the peel strength of the bonded polymers is sufficiently high to cause failure within the PET film (approx. 0.4 N/mm). This film failure is desirable for food packaging as it ensures a strong seal with no leakage. It was found that plasma treatment in a He only plasma exhibited maximum adhesion, causing film failure across the full range of temperatures investigated. This represents a 25 fold increase in peel strength at a heatsealing temperature of 160°C. The effect of O₂ addition into the plasma was also evaluated. It was found that the APET polymer samples treated in a He / O₂ plasma exhibited full adhesion above a heatsealing temperature of 140°C. Below this temperature, an increase in adhesion was observed but it was not of sufficient strength to cause failure of the film. This 20°C difference in heatseal temperature for the addition of O₂ is most likely due to quenching of the plasma leading to a reduction in the reactive species present in the discharge [25].

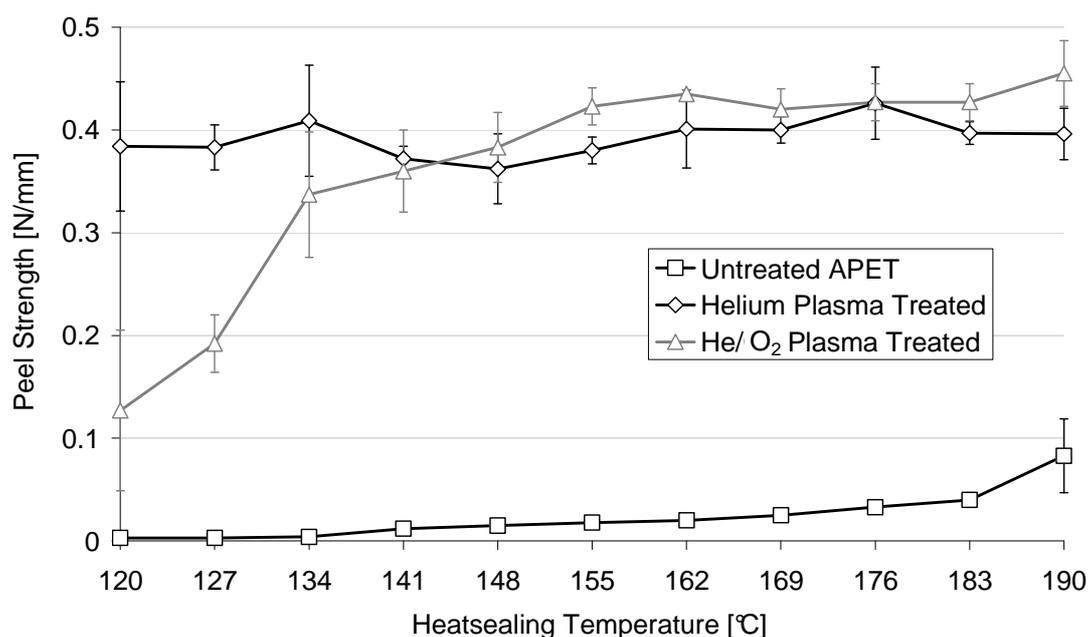


Figure 3: Peel Strength of untreated PET film thermally bonded to plasma treated APET at varied temperature

The surface energy of plasma treated samples was analysed as a function of time after treatment using the contact angle method (Figure 4). The surface energy measured 24 hrs after treatment was found to increase from 40 to approximately 57 mN/m for both He only plasma treatment and He / O₂ plasma treatments. The higher surface energy observed after plasma treatment is associated with a more hydrophilic surface. The surface energy was observed to slowly decrease with time after treatment. This is related to the motion and reorientation of hydrophilic functionalities away from the

surface and towards the bulk of the polymer. This is commonly referred to as hydrophobic recovery [26, 27].

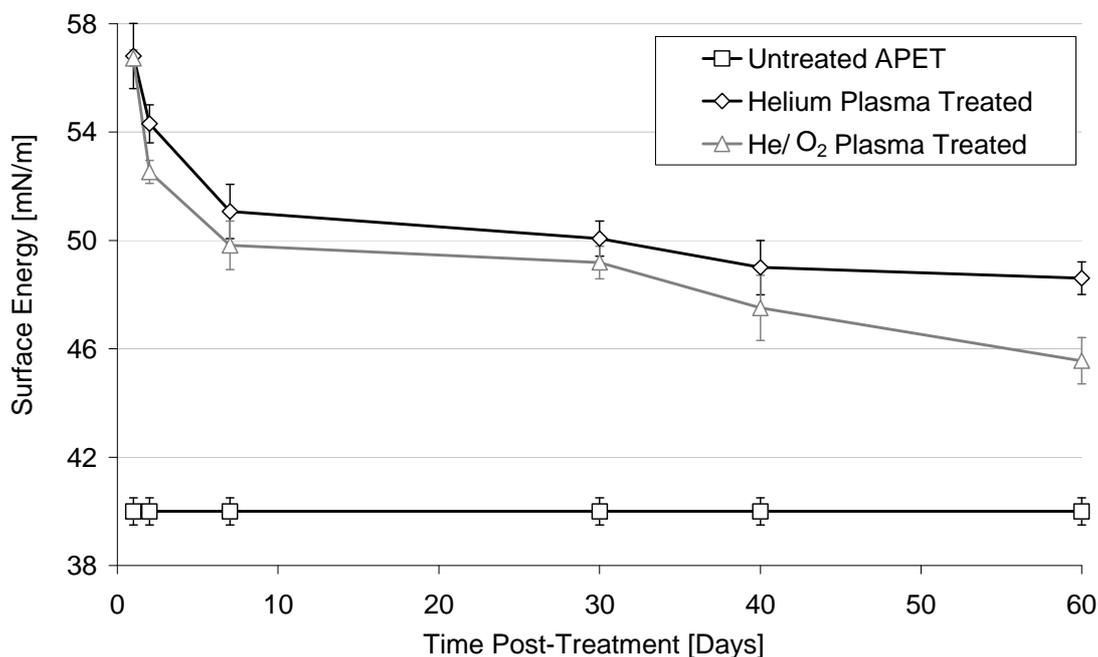


Figure 4: Surface Energy of plasma activated APET as a function of time after plasma treatment

Chemical functionality of the plasma treated APET material

In order to determine the effect of the plasma treatment on the polymer a surface analysis study was carried out. For these trials APET samples were treated using the LabLine system for 125 seconds in a He plasma at 1000 W. x-ray photoelectron spectroscopy (XPS) was used to analyse and compare the surface chemistry of APET polymer before and after exposure to the He plasma. As detailed in Table 1 the elements detected at the surface of the untreated APET were carbon and oxygen. The elements detected at the surface of the APET treated with a He plasma were carbon, oxygen and nitrogen. It was found that subsequent to the He plasma treatment there was an increase in the level of oxygen and nitrogen on the APET polymer surface. As shown in Figure 5 the carbon 1s spectra obtained from the untreated APET sample were fitted and three peaks representative of the functional groups C-OC*=O, C*-OC=O and C-C and a peak representative of a pi-pi* shake up satellite were obtained. These peaks have previously been attributed to these chemical functionalities by Liston, Martinu and Wertheimer [28]. It was found that the pi-pi* shake up satellite had a greater intensity in the carbon 1s spectra acquired from the surface of the plasma treated APET. This pi-pi* shake up satellite is a results of the photoionisation which takes place during XPS analysis and is associated with unsaturated double bonds such as C=C and C=O. It was also noted that the relative intensity of the peak representative of aromatic hydrocarbon decreased after helium (He) plasma treatment. This indicates that chain scission of the APET may have occurred resulting in “dangling” aromatic groups. Three additional peaks were fitted to the carbon 1s spectra acquired from the surface of the plasma treated APET sample. They were representative of amine (C-N), carbonyl (C=O) and anhydride (O=C-O-C=O) functional groups incorporated in the surface by the plasma treatment. There is also a C-Ar & C-Si peak observed. The LabLine systems is also used

extensively for application of silane coatings and argon plasma treatments. Although intensive cleaning is carried out between experiments it is thought that these peaks can be attributed to trace amounts of these products remaining in the system. Both the LabLine and AP4 systems are industrial reel to reel systems where various coatings and treatments could be carried out on the same day. The addition of the both oxygen and nitrogen functional groups to the APET surface is observed to cause an increase in both the surface energy and heatsealability after plasma treatment.

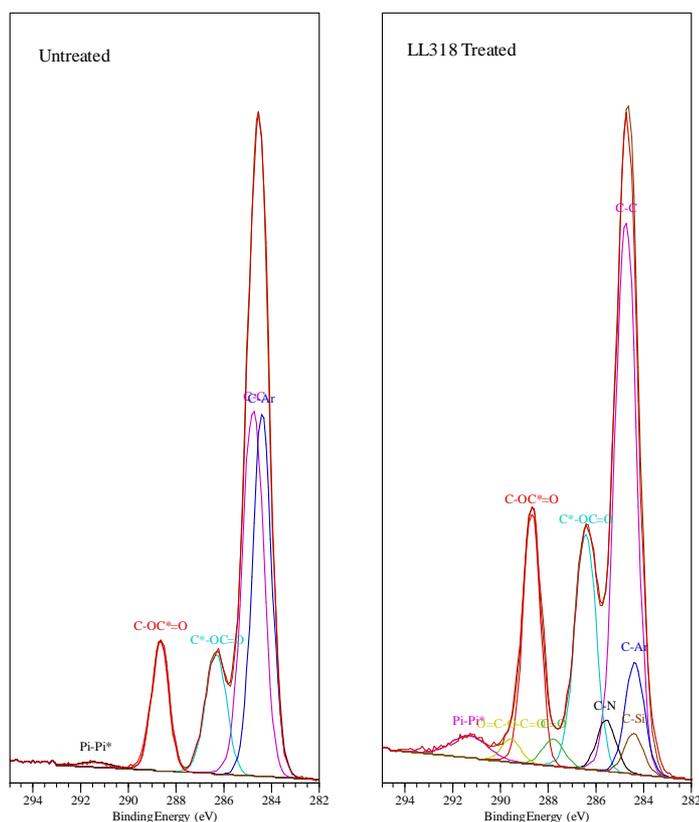


Figure 5: High resolution carbon 1s spectra acquired from the APET samples

The relative amounts of carbon, oxygen and nitrogen detected are shown in Table 1. The error values given are \pm twice the standard deviation. As these treatments were carried out in a He only plasma these results indicate that a sufficient level of O_2 may be present in the LabLine chamber to provide a functional activation without any O_2 being added through the gas feed lines. This could be a result of air diffusing into the chamber through the opening at the bottom of each set of electrodes which is used to pass the polymer material into the system.

Table 1: The relative atomic percentages of elements detected at the surfaces of the APET samples

| | % C | %O | %N |
|---------------------|----------------|----------------|---------------|
| Untreated APET | 82.6 ± 0.3 | 17.4 ± 0.3 | |
| Plasma Treated APET | 68.7 ± 1.3 | 28.6 ± 0.3 | 1.3 ± 0.6 |

Surface morphology of plasma treated APET

The surface morphology and roughness of both the untreated and plasma treated APET polymer was analysed using a CP-II (Veeco) atomic force microscope (AFM). Regions of $1 \times 1 \mu\text{m}$ were scanned and are shown in Figure 6. The roughness values assessed were the average roughness (R_a), root mean square roughness (R_q) and the peak to valley roughness (R_{p-v}).

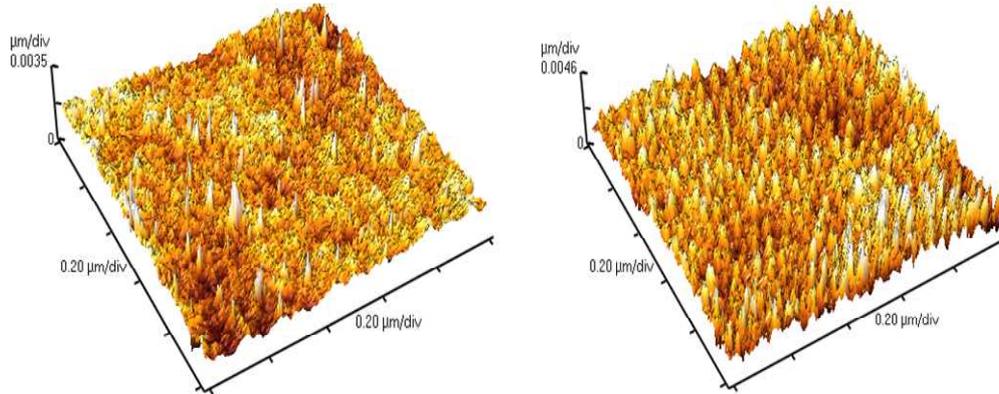


Figure 6: AFM analysis of untreated APET (left) and plasma treated APET (right), LabLine Conditions: 1000W, 125 secs, He plasma

The surface morphology of the APET was observed to change significantly after plasma treatment using the LabLine in a He plasma at 1000 W for 125 secs. The results of roughness measurements of the treated samples are given in Table 2. The error is obtained from standard deviation of 3 surface roughness measurements. It was found that the roughness as measured using peak to valley (R_{p-v}), root mean squared (R_q) and average roughness (R_a) approximately doubles for the plasma treated APET samples. The roughened surface morphology is due to energetic species within the plasma impacting on the polymer surface [29-31].

Table 2: Surface roughness analysis of APET samples using AFM

| | R_{p-v} [nm] | R_q [nm] | R_a [nm] |
|---------------------|----------------|---------------|---------------|
| Untreated APET | 4.7 ± 0.5 | 0.5 ± 0.1 | 0.4 ± 0.1 |
| Plasma Treated APET | 7.0 ± 0.5 | 1.1 ± 0.1 | 0.9 ± 0.1 |

Industrial scale plasma treatment of APET

As was observed in the previous section, pilot scale trials using the LabLine plasma treatment system significantly enhanced the adhesion between APET polymer and the PET top film. In this section larger scale treatment studies were carried out using the AP4 system. The effect of applied plasma power, gas flow and treatment time were investigated in order to optimise the heatseal adhesion. These trials involved thermoforming food trays from 600 mm wide rolls of APET, which were plasma treated using the AP4 system. Thermoforming was carried out using the TFT 780 thermoformer. The surface energy, peel strength and burst pressure of the trays thermoformed from plasma treated APET were tested.

Effect of applied plasma power and throughput speed on the heatsealability of APET

The industrial plasma treatments were carried out using the AP4 system. The applied power was altered between 4000 and 7500 W. The treatment time was varied by increasing the speed at which the APET polymer moves through the electrodes. The treatment conditions used for initial testing are given in Table 3. For these trials only He was used to generate the plasma.

Table 3: Plasma treatment conditions for initial testing

| | <i>Applied plasma power [W]</i> | <i>He flow rate [L/min]</i> | <i>Treatment time [Seconds]</i> |
|-----------|---------------------------------|-----------------------------|---------------------------------|
| Treated A | 4000 | 30 | 23 |
| Treated B | 4000 | 30 | 8 |
| Treated C | 7500 | 30 | 23 |
| Treated D | 7500 | 30 | 8 |

The surface energy of the treated APET polymer was tested using the sessile drop technique and the results are given in Figure 7. As demonstrated in this figure both applied power and treatment time had a significant influence on the treated APET polymer surface energy. The highest surface energy of 52 mN/m 1 day after plasma treatment C was observed for samples passed through the chamber at low speed which corresponds to long treatment times. Samples which were treated for a short time (Treatment B & D) exhibited surface energies of 44 and 48 mN/m for low and high power respectively.

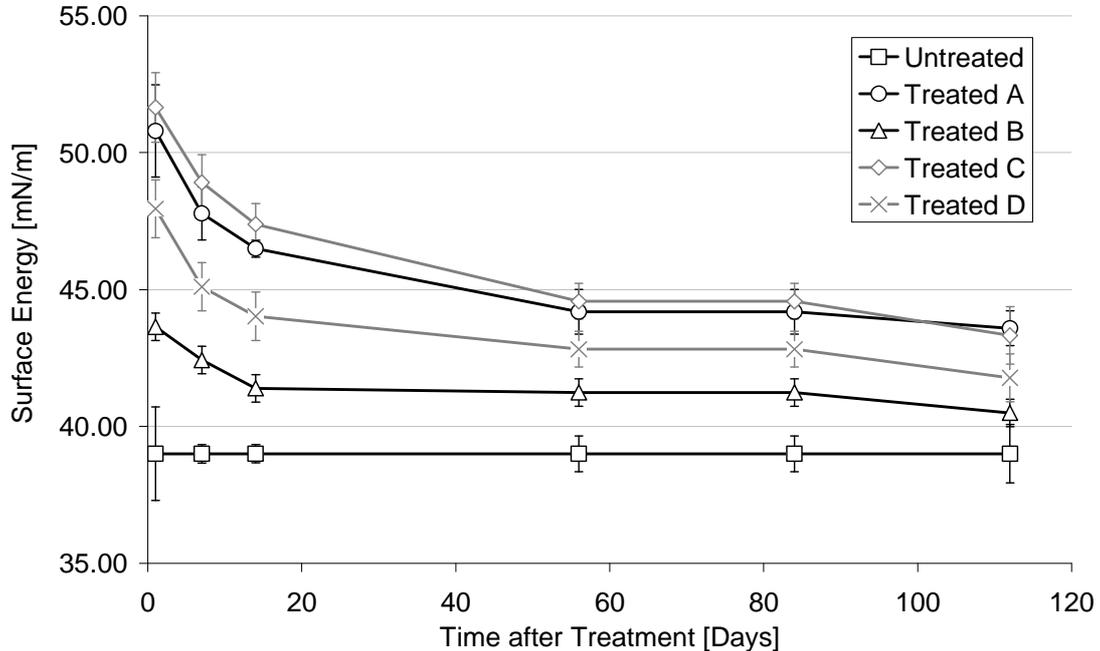


Figure 7: Effect of power and treatment time on surface energy of PET film bonded to APET polymer plasma treated using the AP4 system. See Table 3 for parameters.

The applied power and treatment time were also observed to affect the peel strength of PET film heatsealed to the plasma treated APET (Figure 8). For these tests samples measuring 75 x 25 mm were cut from the plasma treated APET and heatsealed to untreated PET film with a 25 x 25 mm overlap using the HSE 3 heatsealer at a

temperature of 165°C for 1 second. The samples treated at low power and low treatment time (Treatment B) did not exhibit a significant increase in adhesion. All other samples showed a level of adhesion which was sufficient to cause failure within the PET film substrate (approximately 0.4 N/mm). This increased adhesion was monitored up to 16 weeks after treatment. Although most treatments exhibited a large initial increase in heatseal bond strength this increase in adhesion was found to reduce significantly after approximately 60 days in storage, to a value close to that of the untreated APET polymer. This reduction in heatsealability with time after treatment as outlined previously is related to the hydrophobic recovery of the treated polymer surface. It was found that the surface energy measurements (Figure 7) correlated well with the observed peel strengths (Figure 8). Treatments which affected a large increase in surface energy were also observed to cause a large increase in peel strength. The APET samples which were treated for long treatment times (A and C) were observed to provide the highest levels of surface energy and peel strength. The decrease in peel strength observed after 60 days in storage indicates that in an industrial environment, these food trays would have to be sealed within this time period or retreated to ensure that enhanced sealability is present.

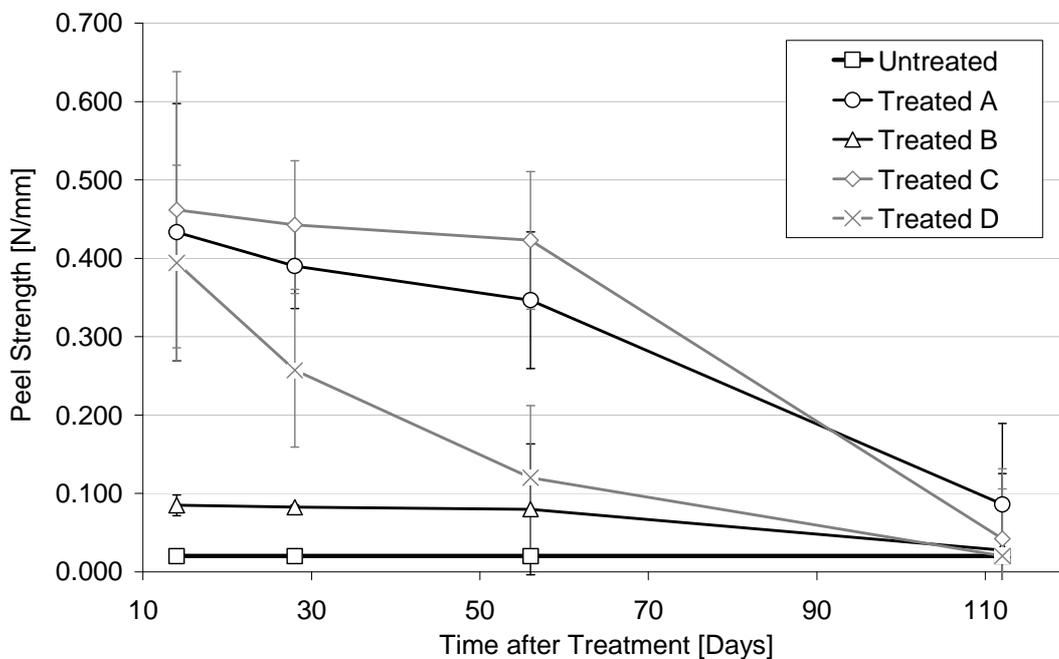


Figure 8: Effect of power and treatment time on peel strength of PET film bonded to APET polymer plasma treated using the AP4 system. See Table 3 for parameters.

Effect of oxygen addition during plasma treatment on the heatsealability of plasma treated APET sheet to PET film

Further industrial trials were carried out to assess the effect of adding O₂ to the He plasma on heatsealing performance of APET plasma treated using the AP4 system. The plasma treatment conditions are given in Table 4. These conditions are the same as those described earlier in Table 3 except for the addition of O₂.

Table 4: Plasma treatment conditions for addition of O₂ tests

| | Applied plasma power [W] | He flow rate [L/min] | O ₂ flow rate [L/min] | Treatment time [Secs] |
|-----------|--------------------------|----------------------|----------------------------------|-----------------------|
| Treated A | 4000 | 30 | 1.5 | 23 |
| Treated B | 4000 | 30 | 0.3 | 8 |
| Treated C | 7500 | 30 | 0.3 | 23 |
| Treated D | 7500 | 30 | 1.5 | 8 |

The surface energy for all samples was observed to increase to between 49 and 56 mN/m after plasma treatment (Figure 9). An increase was observed with O₂ addition for samples treated at low power and treatment time (Treatment B) in comparison to a He only plasma (Figure 7). This demonstrates the importance of O₂ addition to the AP4 system during plasma treatment of APET. Additionally, samples treated under similar conditions in the absence of O₂ showed greater variation in surface energy as seen in Figure 7. The samples treated in He / O₂ plasma also exhibited a difference in their ageing effect. For all He / O₂ treatments the surface energy after 112 days (16 weeks) was above 45 mN/m² (Figure 9) in comparison to He treated APET where all samples exhibited surface energy below 45 mN/m² after 112 days (Figure 7). The increased surface energy for O₂ addition as described earlier is associated with the formation of polar groups on the surface of the polymer [32].

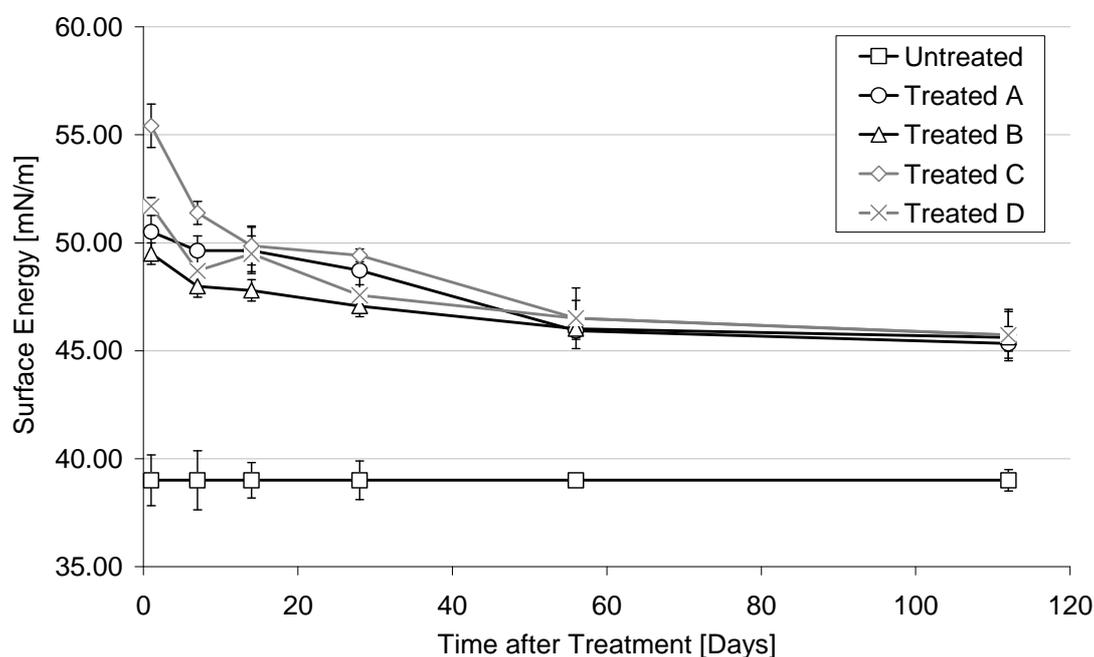


Figure 9: Effect of oxygen addition to the plasma on surface energy of PET film bonded to APET polymer plasma treated using the AP4 system. See Table 4 for parameters.

The peel strength of the APET polymer which was plasma treated in an O₂ containing He plasma discharge is given in Figure 10. It was observed that addition of O₂ to the He plasma increased the thermal bond strength of all samples to a level which caused failure of the PET film (0.4 N/mm), for the range of treatment parameters examined. For these He / O₂ trials the peel strength was found to be insensitive to changes in

applied power, O₂ flow rate and treatment time. The higher peel strength obtained with the He / O₂ plasma compared with the He plasma (Figure 8) demonstrates that the former provides a more robust treatment for enhanced heatsealability. As illustrated in Figure 10, after 60 days storage the peel strength of the treated samples is observed to decrease towards that of the untreated APET. This indicates that the treated material must be either heatsealed or re-treated after 60 days storage for enhanced sealability to be effective. The enhanced performance with oxygen addition in the AP4 system is in contrast to those observed for the LabLine system where O₂ addition was found to quench the He plasma. This may indicate that the AP4 system can ionise O₂ within the He process gas more efficiently than the LabLine system. Due to the failure of the film during testing there is a relatively large strength deviation once the film reaches maximum peel strength. This film failure indicates that a strong bond has been achieved through plasma activation.

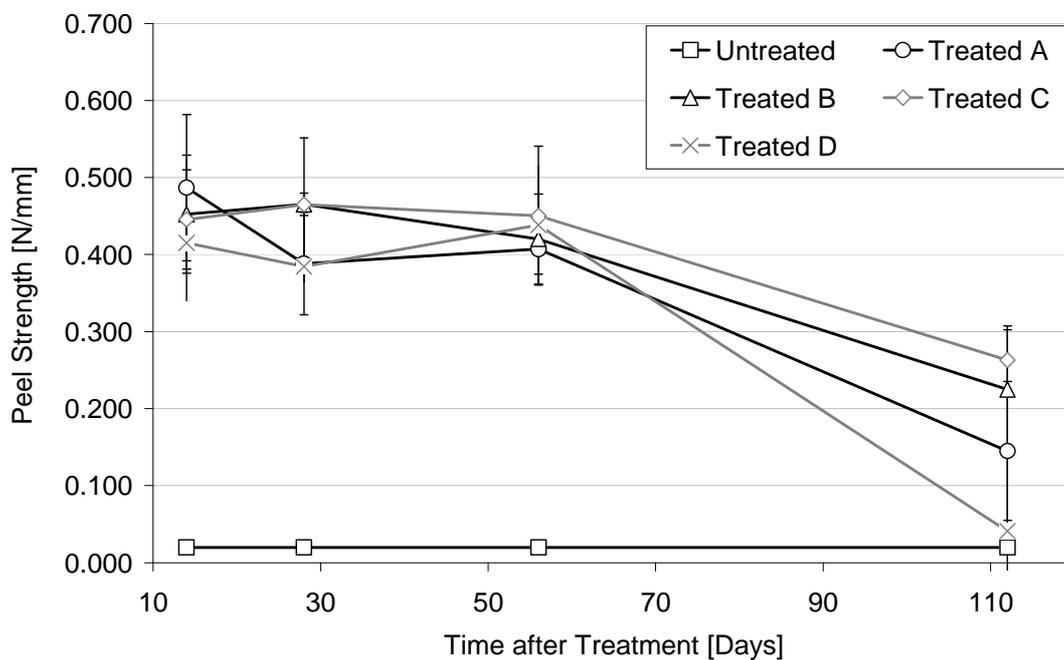


Figure 10: Peel strength of He / O₂ plasma activated PET films bonded to APET (treatment parameters given in Table 4). Note the decrease in bond strength obtained for the ‘aged’ polymer surfaces which were heatsealed after the number of days shown.

Burst pressure of thermoformed food trays

A series of tests were carried out using the three plasma systems described. For the LabLine and AP4 trials sheet material was plasma treated which was subsequently thermoformed in to food trays and burst tested. For the PlasmaStream trials the thermoformed food trays were plasma treated after the tray had been formed. As the PlasmaStream is a jet system it is capable of treating large three dimensional objects whereas the LabLine and AP4 are restricted to the 5 mm electrode gap. The treatment parameters chosen were the optimised conditions for each system. The LabLine was operated at 1000 W in a He only plasma for 125 seconds, the AP4 was powered to 6500 W in a He plasma with 2.5% O₂ for 11.5 seconds, while the PlasmaStream was set to 100 V in a He only plasma, the jet passes 10 times around the sealing surface of the tray at a height of 2 mm and a speed of 10 mm/sec (total treatment time 15 seconds). The untreated APET food trays bonded to the PET film at a burst pressure of 18 kPa. It was found that an increase was observed for all applied plasma

treatments (Figure 11). For the LabLine system and increase up to 28 kPa was observed, while the AP4 system exhibited adhesion up to 25 kPa. The maximum adhesion was observed for the plasma treatments applied using the PlasmaStream system 35 kPa. This increase may be due to the relative intensity of the plasma generated using the jet system and the fact that these treatments are applied after the thermoforming stage in contrast to both the LabLine and AP4 systems. The comparable increase in burst pressure appears to be less pronounced in comparison to the peel strength results however these results should not be directly compared in this manner as the testing technique is different. It should be noted that the PlasmaStream system would not be suitable for use in a production environment due to the batch wise nature of the process. The LabLine and AP4 system in contrast are designed for using in reel-to-reel processing of polymers and could be incorporated into the production line either after extrusion or just before the thermoforming stage.

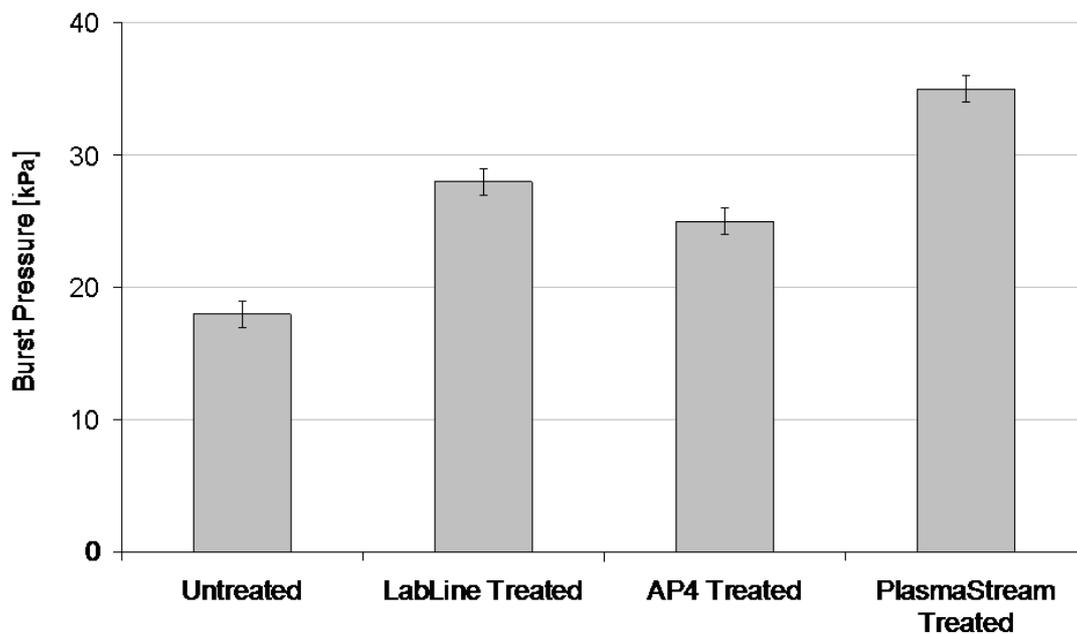


Figure 11: Comparison of the burst pressure obtained for un-activated and plasma activated APET food trays. The plasma pre-treatments were carried out using the LabLine, AP4 and PlasmaStream systems.

CONCLUSIONS

It has been demonstrated that plasma treatment of APET polymer is a viable means of increasing its heatseal properties for the application of food packaging.

- An increase in oxygen and nitrogen containing functionalities was observed after plasma treatment using the LabLine system. The surface roughness was also observed to increase after plasma treatment.

- A 25 fold increase in peel strength was observed for flat samples plasma treated using both the LabLine and AP4 systems. This increase was maintained for up to 60 days after plasma treatment
- Some differences were observed in the optimum plasma processing chemistry between the 0.3M LabLine and the 1.2M AP4 web treatment systems. The optimised heatsealing conditions for the LabLine was found be a He only plasma pre-treatment of the APET. In contrast with the AP4 system enhanced heatsealing performance was found when O₂ was added into the He plasma use for the PET pre-treatment.
- A comparison study of the three systems and their effects on burst pressure was also carried out. It was found that while the PlasmaStream system provided the highest level of adhesion enhancement the LabLine and AP4 system were more suitable to a production environment as they operate in a reel-to-reel manner.

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