

# Surface hybridisation of semi-porous particle foams

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## Abstract

Plasma treatment is a reliable technology for the activation of plastic surfaces. Surface modifications are evaluated with the aid of surface energy measurement, which in turn provides a basis for the derivation of adhesive potential. Sufficient adhesion is a prerequisite for the manufacturing of over-moulded structures.

The work presented in this paper investigates hybrid structures consisting of a semi-porous expanded polypropylene (EPP) particle foam core and an overmoulded layer of polyurethane (PU). Since the level of adhesion between EPP and PU is insufficient for an adequate level of bonding, it is necessary for the surface of the EPP to be modified with the aid of atmospheric plasma treatment (APT). APT and evaluation by means of contact angle measurement both reach their limits when applied to EPP due to the material's thin-walled foam structure and uneven surface. Specially adapted APT parameters are therefore required in order to ensure that adequate bonding between the EPP and the PU is achieved without compromising the physical integrity and visual appearance of the surface.

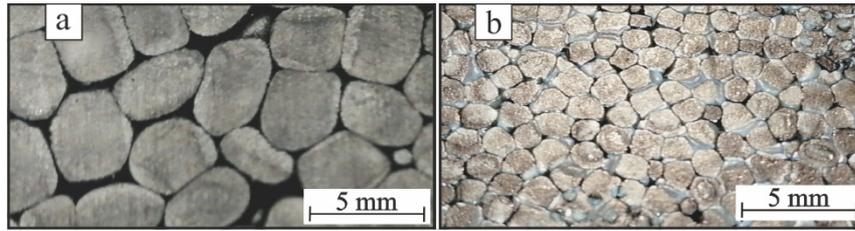
The new approach proposed in this paper uses surface energy determination on both homogenised and semi-porous EPP in order to analyse the transferability of plasma surface modification in EPP. Within the final step the relationship between surface activation, contact angle and adhesion was demonstrated using the floating roller peel test.

**Keywords:** *particle foam, expanded polypropylene (EPP), surface treatment, atmospheric plasma treatment (APT), polyurethane (PU), contact angle, surface energy, floating roller peel test*

## 1 Introduction

Particle foams are a well-established industrial material due to the potential offered by their hollow lightweight design and shaping versatility. The spread of the use of particle foams such as EPP is also attributable to their good insulation properties, high energy absorption rates and individually adjustable densities [1].

The manufacturing process for standard EPP generally results in a surface quality that is insufficient for Class-A surfaces (fig. 1a and 1b). Additional surface layers such as coating films are therefore typically required in order to seal and embellish the surface. In view of these developments, over-moulding of components with clear polyurethane systems represents an opportunity to protect the surface while also maintaining the desired appearance of the individualised structure. Pre-treatment of the low-energy surface of the EPP is nevertheless required in order to achieve sufficient chemical bonding between the particle foam and the polyurethane.

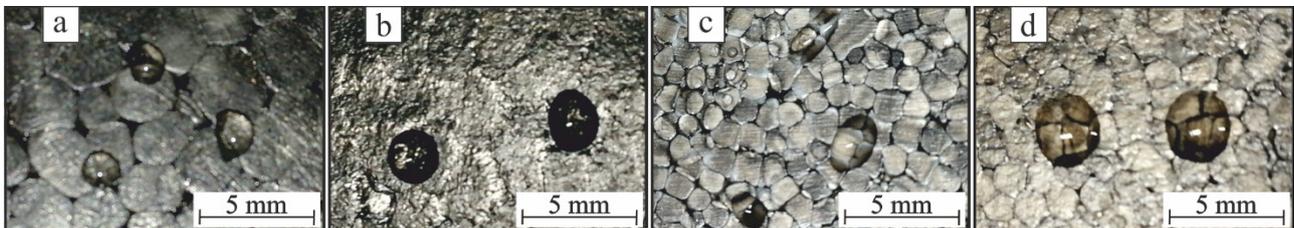


**Figure 1:** Surface of two particle foams: a) 50 kg/m<sup>3</sup>; b) 100 kg/m<sup>3</sup>

Plasma pre-treatment is a technologically and economically efficient means of increasing the strength of the bond between the surface and the coating [2]. A number of studies have already demonstrated the improvements in adhesion achievable by adjusting a variety of APT parameters. The evaluation of those improvements involves the use of surface energy measurements, mechanical pull-off tests and shear tests [3, 4]. Sufficient bonding with low-energy surfaces such as polypropylene (PP) or polyethylene (PE) – both of which are components of EPP – is dependent on a minimum surface energy of 38 mJ/m<sup>2</sup> [2]. Non-expanded PP and PE have surface energies of 31-32 mJ/m<sup>2</sup> [6], while the surface energy of EPP is approx. 23 mJ/m<sup>2</sup> [3]. The actual difference in surface energy depends on the composition of the particle foam in question, and in particular any additives used and their effect on the expansion process. It is assumed that thin-walled pore structures react to APT in a different way.

Plasma parameters characterised by high energy input, slow feeding rates and close nozzle position lead to visible and physical damage to the surface and are therefore unsuitable for the pre-treatment of EPP. Standard, non-material-specific parameter sets can also be disregarded as they do not exploit opportunities to use material-specific properties to achieve optimum bonding strength. As such, it is necessary to identify adapted plasma parameters that maximise adhesive quality without affecting the appearance of the foam. A qualitative means of using surface energy measurement to characterise the impact of APT is therefore required.

The measurement of the surface energy of EPP is difficult due to the pores and unevenness of its surface. Surface energy measurements therefore need to be carried out in sections where surface particles are large and closed. Despite being a technical possibility, this approach is prone to failure in the case of EPP with a low density of around 50 kg/m<sup>3</sup> since surface conditions lead to an uneven formation of the test liquid droplets (fig. 2a) and amplify the amount of experimental effort required. An increase in density (to 100 kg/m<sup>3</sup>) nevertheless results in a decrease in particle size and an increase in the micro pore content of the surface, thus preventing reproducible measurements (fig. 2c).



**Figure 2:** Drop shape on semi-porous surfaces a) and c) and homogenised surfaces b) and d)

A new approach to the determination of the surface energy of EPP was investigated with a view to ensuring reliable, efficient measurements. It was found that the surface of the particle foams could be homogenised by means of a gentle compression process without causing any significant change in density. The homogenised surfaces allowed the execution of the required measurements in accordance with DIN EN 828 (fig. 2b and 2d).

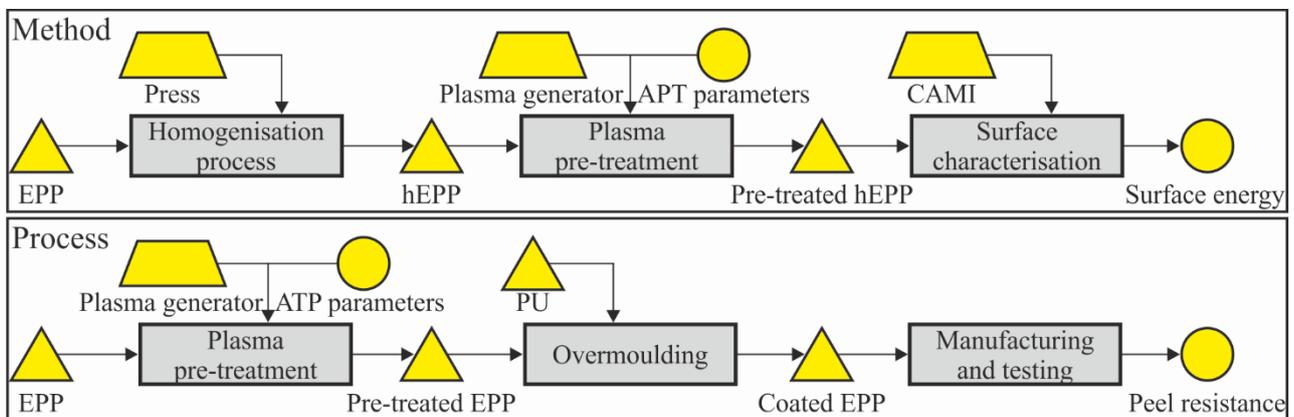
This paper describes a method for the indirect determination of the surface energies of particle foams and discusses associated possibilities and limitations.

## 2 Investigation

### 2.1 Materials and procedure

The low-density EPP “ARPRO A5635” (ARPRO) (fig. 1a) was selected for the experimental studies. The higher-density EPP “ASAHI KASEI Sunforce” (Sunforce) (fig. 1b) was also investigated in order to assess the transferability of the new method. Sunforce consists of a mixture of PP, PE and polyphenylene ether (PPE), whereas ARPRO only contains PP and PE. The two materials are referred to jointly as “EPP” in the following.

The surface of the EPP was homogenised (hEPP), pre-treated using APT and characterised with the aid of a contact angle measurement instrument (CAMI). The results were used to select the best APT parameters for the pre-treatment of the EPP. The subsequent overmoulding of the EPP with PU was followed by the determination of peel resistance using floating roller peel tests (fig. 3).



**Figure 3:** General methodological procedure and process diagram

### 2.2 Homogenisation process

A 4-stage compression process was used to achieve a plane EPP surface. The compression plates and the surface of the EPP were cleaned and degreased using isopropanol in order to avoid any contamination during the compression process. Each homogenisation process saw one pair of test specimens (dimensions: 120x80x16 mm) heated to 150 °C by the heated plates and compressed at a constant load of 2 kN for 1 minute. Homogenisation was carried out with the aid of a RUCKS laboratory press. The resultant surface was even and exhibited a significant 95 % reduction in bubbles and porous defects (fig. 2b and 2c).

One of the effects of surface homogenisation was a reduction in specimen thickness equivalent to the increase in local near surface density. The effect was greater in the case of ARPRO (final thickness: 8 mm) than in the case of Sunforce (final thickness: 13 mm).

### 2.3 Surface treatment

The selection of the APT parameters for the investigation was based on a literature review [7], previous experience gained at the ILK [8, 9], process window recommendations given by Plasmatreat GmbH, technical capabilities and economic factors. Reproducible results were achieved by setting up a robot-guided apparatus for the plasma treatment process. Generator FG5001 was used in combination with nozzle RD1004. Focus on a single influencing factor was ensured by only varying the distance between the nozzle and the surface. Frequency (21 kHz), plasma cycle time (100 %),

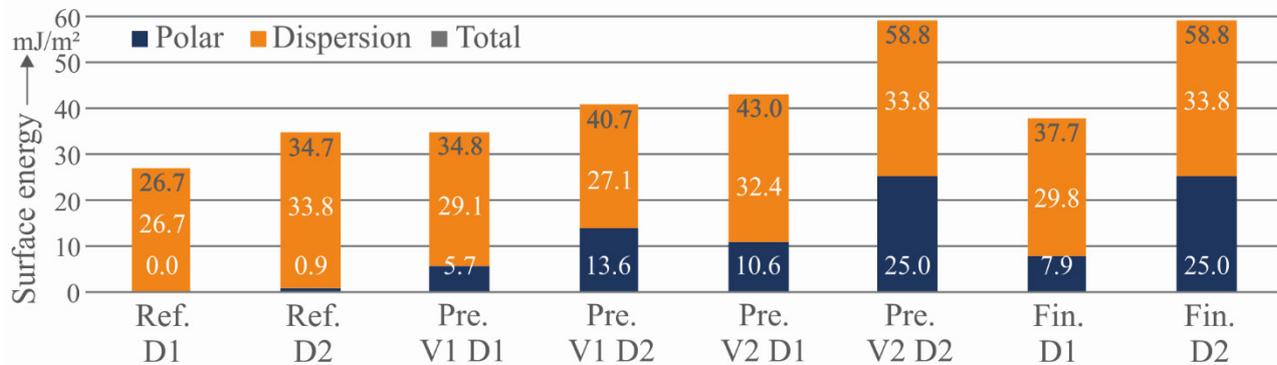
ionisation gas flow rate (45 l/min), nozzle rotation speed (2600 min<sup>-1</sup>) and movement speed (10 m/min) remained constant. The various sets of APT parameters applied are listed in table 1. All parameters are subdivided into preselection parameters for the measurement of the surface energy of the homogenised substrate (Pre.) and final parameters for the floating roller peel tests (Fin.). The reference surface energy without APT was also determined (ARPRO: Ref. D1; Sunforce: Ref. D2).

**Table 1:** APT sets

	Pre. V1D1	Pre. V2D1	Pre. V1D2	Pre. V2D2	Fin. D1	Fin. D2
Distance [mm]	18	6	18	6	16	6
Density [kg/m <sup>3</sup> ]	50	50	100	100	50	100

## 2.4 Surface characterisation

The effect of APT was determined with the aid of contact angle measurements using the “Dataphysics OCA 15 EC” contact angle measuring instrument. Each test liquid contained different polar and dispersion components (water, diiodomethane and ethylene glycol). 10 drops of each liquid were applied to the APT-treated surface. The impact of drop settling on the measurement was minimised by measuring the contact angle 10 seconds after drop application. The polar and dispersion components of the hEPP surfaces were calculated using the Owens, Wendt [10], Rabel [11] and Kaelble [12] model and compared with the Young equation. The results are shown in figure 4.



**Figure 4:** Surface energy and polar/dispersion components for different APT parameters

The results show the effect of APT as a function of the respective increase in surface energy. The increase in polar components shows that better results can be achieved by reducing the distance between the nozzle and the material.

The APT parameters for Sunforce can be transferred directly from hEPP to EPP. It is therefore assumed that surface energy measurements can also be transferred. This verifies the suitability of the method for high-density EPP.

ARPRO behaves differently, with the best set of parameters (Pre. V2D1) causing unacceptable damage to the surface of the EPP. It was for this reason that nozzle distance was increased until no surface damage occurred (Fin. D1). The resultant difference of 10 mm dictates that the transferability of results between hEPP and EPP is significantly lower in this case. This lack of transferability is attributable to the effect of compression during homogenisation. Higher material densities enable a reduction in the distance between the nozzle and the surface during APT, thus leading to a higher surface energy level.

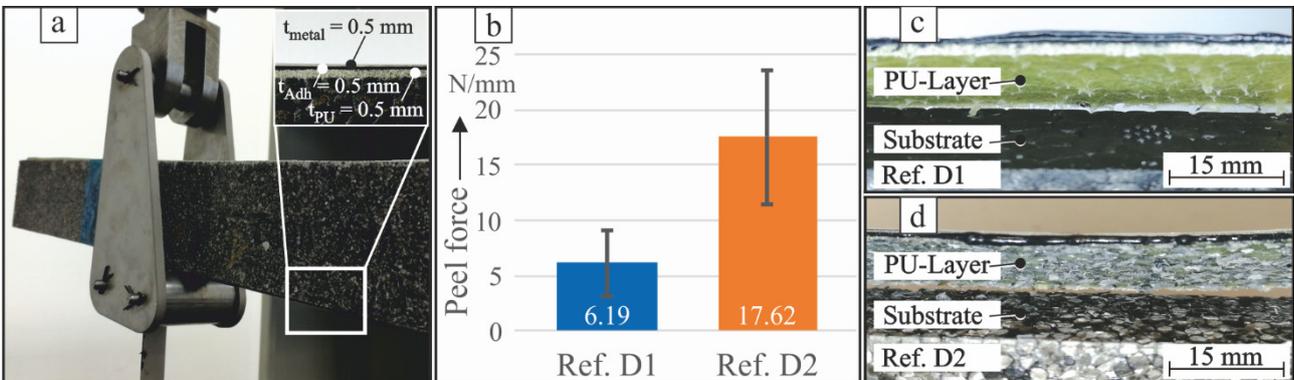
## 2.5 Specimen manufacturing, floating roller peel tests

Floating roller peel tests were preceded by the overmoulding of a PU layer onto all specimens within an hour after APT. This approach ensures optimum bonding due to the dependence of plasma pre-treatment on ageing time [7]. The surface of the specimens was cleaned and degreased using isopropanol prior to overmoulding. The Rühl Puromer GmbH product “puropreg 185 LT IT” was selected as the overmoulding material. The coating was hand mixed before being applied to the surface.

For the subsequent curing process the specimens were stored at room temperature for 7 days. The PU layer was then sanded and bonded to a metal peel strip using a 2-component epoxy adhesive (Scotch-Welt DP490). A diagram of the bonding structure is provided in figure 5a. The width of the test specimen was set at 16 mm in order to reflect available manufacturing capabilities.

Uniform test conditions were ensured by storing all specimens in standard climatic conditions (23 °C, 50 % relative humidity) for a period of 24 hours prior to the test. The test itself was performed on a Zwick/Roell 1465 universal testing machine. Forces were measured over a distance of 200 mm at a travel speed of 100 mm/min and a preload of 5 N.

Examples of the results of the floating roller peel test are provided in figure 5b. It is noticeable that the materials tested performed differently depending on their chemical composition and mechanical surface structure. Sunforce failed at a peeling force of 17.62 N/mm ( $\sigma = 6.01$ ), whereas the lower-density ARPRO failed at 6.19 N/mm ( $\sigma = 3.03$ ). Monitoring of failure behaviour during the peeling process found adhesion delamination to occur between the PU and the EPP throughout the peel distance (fig. 5c and 5d). As the particle foams consist mainly of PP and PE, it can be assumed that surface texture was a significant factor in the difference between the peel forces observed. The higher number of smaller pores and greater unevenness that characterise the surface of the Sunforce material are conducive to mechanical undercutting and the strengthening of the bond between EPP and PU.



**Figure 5:** a) Test set up; b) Examples of the results of the floating roller peel test (carried out in accordance with DIN EN 1464); c), d) Failure pattern

Contrary to expectations, it was the consistent failure of the adhesive bond between the PU layer and the metal peel strip – and not the adhesive bond between the EPP and the PU – that caused all APT-treated specimens to fail the floating roller peel test. The increase in the strength of the bond between the PU and the APT-treated EPP indicates a shift in the fracture pattern. Although the accurate determination of the peel resistance of APT-treated EPP requires a specially adapted test setup, the variation of the adhesive used is not advisable due to the fact that the selected adhesive system possesses very good mechanical properties. It may be possible to improve the test setup by embedding the metal peel strip directly into the PU layer. Further investigation into the process flow and interaction mechanisms is nevertheless required.

### 3 Conclusion

The surface energy of semi-porous EPP is difficult to determine using contact angle measurements as described in DIN EN 828. A new approach was therefore investigated. In the case of high-density EPP in particular, the method under investigation proved to be a reliable and reproducible means of determining surface energy. It was possible to transfer the APT parameters for the Sunforce material directly from the homogeneous test surface to the EPP surface. The method therefore appears to be an adequate means of determining the surface energy of homogenised surfaces at this density.

In the case of the lower-density EPP, it was necessary to adjust the plasma parameters for the homogenised surface in order to prevent the surface from being burned by the plasma jet. It can be assumed that this issue is attributable to the compaction of the surface during the compression process. As a result, it may be possible to apply more intensive pre-treatment parameters without damaging the surface. This assumption is supported by the very large deviation from the parameters that can actually be applied.

Future work in this field needs to provide further evidence of the suitability of the method at hand by testing it in combination with particle foams of a variety of densities. The homogenisation process also needs to be improved in order to minimise the compression effect.

A significant difference in the peel resistance of the two material variants was observed. The fact that the base material was identical means that chemical bonding can be ruled out as a potential cause of the differences in their adhesive properties. It can be assumed that the cellular structure of an expanded plastic foam surface offers an advantage where bonding strength is concerned. An increase in density leads to a greater number of particle interspaces in the surface, which in turn results in an increase in peel resistance due to the higher degree of mechanical micro interlocking.

The pre-treatment of EPP using APT facilitates enhanced adhesion to PU systems. Despite the use of a variety of different adhesive systems, the consistent failure of the adhesive bond between the metal peel strip and the PU layer prevented the execution of any adhesion tests using the test setup described in this paper. It may be possible to eliminate this issue by embedding the metal peel strip directly into the PU layer.

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