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A novel approach for an in-line atmospheric plasma-treatment of polymers and reinforcements during extrusion

A novel approach for an in-line atmospheric plasma-treatment of composites or blends during extrusion processing using a twin screw extruder is reported. The atmospheric plasma-treatment takes place in a partially filled zone of the twin screw extruder. Exemplarily, a glass fibre-reinforced polypropylene (PP) composite and a polypropylene-polyamide 6-blend (PP-PA6-blend) are processed using the in-line atmospheric plasma treatment process. For both systems a slight increase of Young's modulus and a slight decrease of the elongation at break are observed, while the tensile strength and the notched impact strength of the glass fibre reinforced polypropylene composites are barely affected. The Charpy notched impact strength of the PP-PA6-blend is improved by almost 43 % from 22.5 kJ/m² to 32.1 kJ/m² by applying the in-line atmospheric plasma-treatment process.

Funktionalisierung von Polymeren und Füllstoffen mittels Atmosphärendruck-Plasmen während der Compoundierung

Gegenstand dieser Untersuchung ist ein neuer Ansatz zur Funktionalisierung von Kompositen und Blends mithilfe eines Atmosphärendruck-Plasmas während der Aufbereitung. Die Funktionalisierung erfolgt dabei in einer teilgefüllten Zone eines Doppelschneckenextruders. Es wurden ein Glasfaser verstärktes Polypropylen (PP) und ein Polypropylen-Polyamid6-Blend (PP-PA6-Blend) unter Verwendung der neuen Methode hergestellt. Für die beiden untersuchten Systeme sind ein geringer Anstieg des E-Moduls und eine geringe Reduktion der Bruchdehnung zu beobachten, während die Zugfestigkeit und die Kerbschlagzähigkeit des Glasfaser verstärkten PP nur geringfügig beeinflusst wird. Die Kerbschlagzähigkeit des PP-PA6-Blends wird jedoch um 46 % von 22,5 kJ/m² auf 32,1 kJ/m² durch die Funktionalisierung mittels Atmosphärendruckplasmas gesteigert.

A novel approach for an in-line atmospheric plasma-treatment of polymers and reinforcements during extrusion

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1 INTRODUCTION

Melt-mixing two or more components, of which at least one is a polymer and the others being either different polymers, reinforcements or fillers, is a key task in polymer processing, generating polymer-blends in the case of mixing different polymers and polymer-composites, when fillers are added to one or more polymers. In general, the physical and chemical interactions between the polymers or the polymers and fillers can be related to the morphology and the stability of the resulting system, which in turn determine the final properties [1]. Thus, for both polymer-composites and polymer-blends the interface, where these physical and chemical interactions occur, plays a crucial role for the final properties. However, due to the literally infinite number of combinations of polymers and/or polymers and fillers different challenges arise, when mixing two (or more) components. Hence, in scientific research and in industry different strategies are developed to meet these challenges, resulting in numerous publications, which report about interfaces of polymer-blends and polymer-composites and for example how to promote wetting of fillers, adhesion and compatibility of different components or how to avoid coalescence of polymer-blends. Table 1 compiles a selection of common strategies to improve interfacial interaction between polymers and fillers by a treatment of the filler [2].

Another strategy to treat fillers prior to melt compounding is applying a plasma-treatment. Different plasma treatments ranging from treatments to increase the surface energy of the fillers [15] to grafting molecules on the filler surface [16-20] are reported in literature. These treatments are studied for a variety of fillers such as carbon fibres [16], calcium carbonate [16], aramide fibres [11], glass fibres [15,17,18,21], polyethylene terephthalate fibres in polymethyl methacrylate [22] or carbon nanotubes and carbon nanofibres [19,20,23]. Plasma treatments for natural fibres are summarized by Kalia et al. [24]. However, all filler treatment strategies have to be carried out in a separate step, before the filler is mixed with the polymer, which is time-consuming and expensive for industrial applications. Another possibility offers the addition of expensive compatibilizing additives such as maleic anhydride grafted polymers during the melt compounding step [25,26].

strategy	**typical fillers**	**reference**
thermal treatment	talc, silica	[3]
surface oxidation	carbon fibres	[4]
stearic acid treatment	calcium carbonate	[5]
isocyanate treatment	kaolin	[6]
silane treatment	silica, natural fibres, glass fibres, kaolin	[7-8]
titanate treatment	kaolin calcium carbonate	[9-10]
coating	aramide fibre carbon black	[11-13]
grafting	calcium carbonate carbon black	[14]

Table 1: Selection of common strategies to improve interfacial interaction between polymers and fillers

Regarding polymer-blends, it is agreed upon, that a significant number of polymer blends is immiscible, due to the unfavourable interaction between molecular segments of the polymer-blend components [27]. Thus, interfacially active copolymers are added [27-38] or formed in situ during processing by an interfacial reaction of added functionalized polymeric components [27,38-42]. The aim of this interfacial compatibilization is to lower interfacial tension, to decrease the dispersed phase domain size, to hinder coalescence, to improve adhesion and to stabilize the morphology [38,43-46]. Additionally, surfaces (and interfaces) of (solid) polymers can be modified by plasma treatments either to graft molecules onto the polymer [56-58] or to increase the surface energy and the hydrophilicity [50]. But until today these compatibilisation and treatment strategies for polymers and polymer-blends either have to be carried out in a separate processing step, involve an expensive copolymer or a chemical reaction which has to be controlled during processing.

Concluding, in polymer-composite and polymer-blend processing there is a need for an in-line treatment strategy, which can be easily controlled, while it is economically reasonable and applicable to a wide range of polymers and fillers. Thus, this technology offers a novel approach to meet the above named criteria by applying an in-line atmospheric plasma treatment of polymers and fillers during extrusion processing.

2 EXPERIMENTAL SECTION

2.1 Materials

Two different material combinations were used to investigate the in-line atmospheric plasma treatment during extrusion processing: A glass fibre reinforced polypropylene (PP) and a polypropylene-polyamide 6-blend (PP-PA6-blend). In both cases a PP homo-polymer type HD120MO of Borealis AG, Vienna, Austria, with a density of 0.908 g/cm³ and a melt flow rate of 8 g/10 min, measured at 230 °C with a weight of 2.16 kg, was used. The Young's modulus and the tensile strength of the polypropylene are 1500 MPa and 33.5 MPa, respectively, while the Charpy notched impact strength accounts to 4 kJ/m² (determined according to ISO 179-1eA). The PP was melt-compounded with either 30 wt.-% glass fibres or 50 wt% PA6. The glass fibres type Chopvantage HP3299 of PPG Industries, Pittsburgh, Pennsylvania, USA, have an average diameter of 14 µm and are cut to a length of 4.5 mm. To improve the compatibility to polypropylene the glass fibres are prepared with a silane-sizing by the manufacturer. The PA type Durethan B30S of Lanxess AG, Cologne, Germany, features a density of 1.14 g/cm³. The Young's modulus of the PA6 is 3000 MPa when dried and 1000 MPa in a conditioned state, while the tensile strength is 80 MPa (dried) and 40 MPa (conditioned), respectively. A Charpy notched impact strength of 10 kJ/m² when dried and 20 kJ/m² when conditioned.

2.2 In-line atmospheric plasma-treatment during extrusion

In general, the materials were prepared using a co-rotating, intermeshing twin screw extruder type ZSK 18 MEGAlab of Coperion GmbH, Stuttgart, Germany. The screw diameter was 18 mm and the length-to-diameter-ratio was 45. For the glass fibre reinforced PP composites, the PP was fed in the main hopper, while the glass fibres were added in the 4th barrel element (14 L/D) using a side feeder. To prepare the PP-PA6-blends a mixture of both polymers consisting of 50 wt.-% PP and 50 wt.-% PA6 was fed in the main hopper. In both cases the materials were metered by suitable gravimetric dosing systems. The materials were mixed and degassed before exiting the extruder as strands. The strands were quenched in a water bath and pelletized by a strand pelletizer. However, to apply an in-line atmospheric plasma treatment the screw configuration of the twin screw extruder was set-up to be partially filled in the 8th barrel element (26 L/D) using conveying elements and inserting a round plasma nozzle into this barrel opening, figure 1.

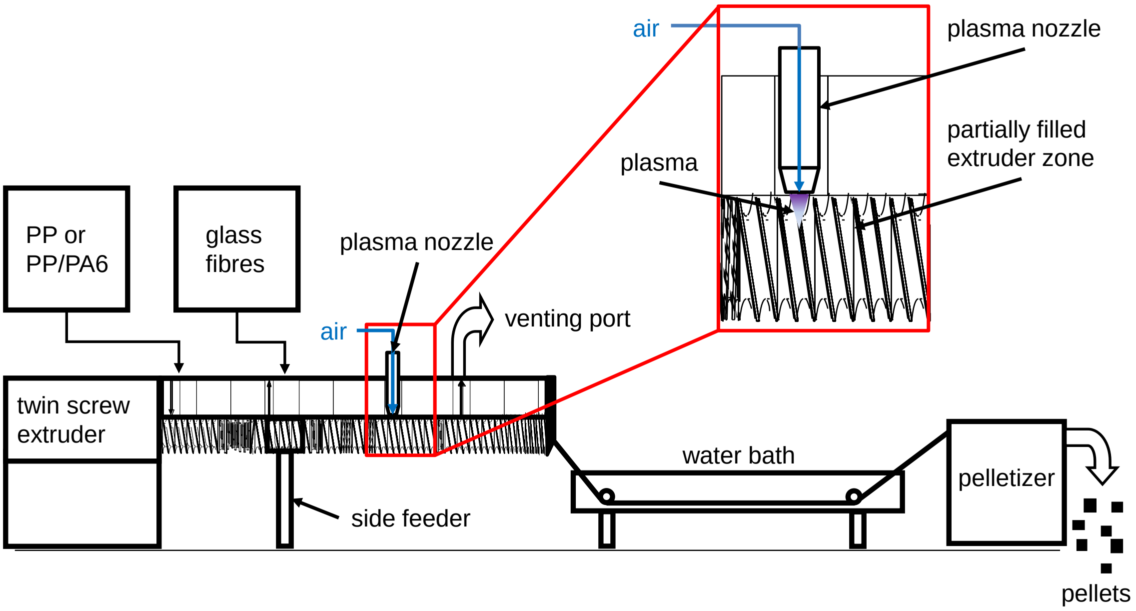

Figure 1: *Schematic of the in-line atmospheric plasma treatment during a continuous melt compounding process using a co-rotating intermeshing twin screw extruder.*

The inserted plasma nozzle is attached to an atmospheric plasma processor type PlasmaBeam of Diener electronic GmbH & Co. KG, Ebhausen, Germany. The plasma of the jet was driven by a generator at 300 W and a frequency of 20 kHz. The jet itself had a length of 270 mm and a diameter of 32 mm. The outlet for the plasma had a diameter of 2 mm. Depending on the processing conditions the treatment area was about 5 to 12 mm. Air at an input pressure of 5 bar was used as process gas.

This way it was possible to realize an in-line atmospheric plasma treatment during a continuous extrusion process.

2.3 Analysis

After the melt-compounding step, the materials are further processed to tensile and notched impact test specimens by injection moulding using an injection moulding machine type Allrounder 370 A 600 - 170/170 of Arburg GmbH & Co. KG, Loßburg, Germany. The processing parameters were kept constant to maintain the comparability of the composites prepared with and without the in-line atmospheric plasma treatment. Young’s modulus, tensile strength and elongation at break were determined according to ISO 527 using a tensile testing machine type Z100 of Zwick GmbH & Co. KG, Ulm, Germany. The notched impact strength was evaluated according to ISO 179 using a pendulum of Zwick GmbH & Co. KG, Ulm, Germany. Before testing, all test specimens were stored for seven days at 23 °C and 50 % relative humidity. Additionally, the morphology of the samples, which were embedded in resin, was investigated using optical microscopy.

3 RESULTS AND DISCUSSION

Even though the stress-strain-curves do not reveal extensive differences of the untreated materials and the materials, which were treated with the atmospheric plasma during melt compounding, slight differences of Young’s modulus, tensile strength and elongation at break can be observed. Concerning the Charpy notched impact strength values quite similar results are observed for the treated and untreated glass fibre reinforced PP composites. However, for the PP-PA6-blends, a large increase of the Charpy notched impact strength is measured, when the in-line atmospheric plasma treatment process is used during extrusion. Table 2 compares the mechanical properties of treated and untreated materials.

Material	**Young’s modulus [MPa]**	**Tensile strength [MPa]**	**Elongation at break [%]**	**Charpy notched impact strength [kJ/m²]**
untreated glass fibre reinforced PP-composite	6848 ± 237	75 ± 1	1.8 ± 0.1	15.4 ± 0.8
treated glass fibre reinforced PP-composite	6936 ± 204	72 ± 4	1.5 ± 0.1	13.8 ± 0.8
untreated PP-PA6-blend	1682 ± 72	32 ± 2	5.5 ± 1.0	22.5 ± 3.5
treated PP-PA6-blend	1854 ± 49	34 ± 1	4.5 ± 0.4	32.1 ± 4.5

Table 2: Comparison of the mechanical properties of the untreated materials and the materials treated with the atmospheric plasma during melt compounding

The in-line atmospheric plasma treatment has only a minor effect on the glass fibre reinforced PP, because the glass fibres are already coated with silane to improve the compatibility to polypropylene. For the PP-PA6-blend the in-line atmospheric plasma treatment leads to a slight increase of the mechanical properties Young’s modulus and tensile strength, which are determined under quasi-static conditions in a tensile test. A much more pronounced improvement of the notched impact strength is observed under sudden stress, which can be attributed to the in-line atmospheric plasma treatment:

To do a statistical analysis of the significance of the effects, a confidence range of the difference δ is determined by equation 1 using the experimental data of PP-PA6-blend of table 2.

$$\bar{d} - t \cdot s_{\bar{d}} \leq \delta \leq \bar{d} + t \cdot s_{\bar{d}} \qquad \text{eq. 1}$$

The difference between two sample means is $\bar{d}$. $s_{\bar{d}}$ is equate to the standard deviation of the effect and t depends on the selected confidence level. s is equivalent to the standard deviation of the single values. N is the scope of experiments.

$$\bar{d} = \bar{y}_2 - \bar{y}_1 \qquad \text{eq. 2}$$

$$s_{\bar{d}} = \sqrt{\frac{4}{N} s^2} \qquad \text{eq. 3}$$

$$s^2 = \frac{s_1^2 + s_2^2}{2} \qquad \text{eq. 4}$$

If the confidence range of the difference δ at a 99 % confidence level does not include the value 0, meaning no change of sign, the influence of the examined parameter is significant [51].

Young's modulus **[MPa]**	**Tensile strength** **[MPa]**	**Elongation at break** **[%]**	**Charpy notched impact strength** **[kJ/m²]**
92.7 ≤ δ ≤ 251.3	-0.04 ≤ δ ≤ 4.04	-1.98 ≤ δ ≤ -0.02	4.41 ≤ δ ≤ 18.79
significant	not significant	significant	significant

Table 3: Calculation of confidence range of the difference for the treated/untreated PP-PA6-blend (t=2.878 [52], N=20)

A comparison of the morphology of the untreated PP-PA6-blend to the morphology of the PP-PA6-blend, which was treated with the in-line plasma-treatment during the melt compounding process, shows a significant difference in the morphology, figure 2.

The microscopy images reveal that the PP-PA6-blend have a matrix-dispersed phase-morphology, in which the PA6 is the dispersed phase. The plasma treatment during the extrusion process leads to smaller domains of the dispersed phase, by decreasing the interfacial tension and thus enhancing miscibility and adhesion. The smaller PA6-domains and the increased interfacial interaction between the polymers efficiently toughen the PP-matrix. Hence, the notched impact strength of the PP-PA6-blend can be improved without sacrificing Young's modulus and the tensile strength. Zeng et al. and Bai et al.

observe a similar behaviour of PP-PA6-blends, when using a polyethylene-octene elastomer grafted with maleic anhydride [53,54]. However, the increase of the impact strength is attributed to introducing the polyethylene-octene elastomer grafted with maleic anhydride into the PP-PA6-blend, which coats the PA6 particles and also remains as small particles in the PP-matrix. The dissipation of the impact energy is described by Bai et al. as follows: Isolated polyethylene-octene elastomer grafted maleic anhydride particles play a small but significant role in either arresting the cracks or at least reducing their propagation rate. Additionally, the high adhesion of the interfacial layer avoids early decohesion at the polyethylene-octene elastomer grafted maleic anhydride interphase between the polypropylene matrix and the polyamide 6 particles, and is later capable of cavitation. Furthermore, the ellipsoidal geometry of the particles improves somehow the impact resistance thanks to its favourable orientation perpendicular to the crack propagation direction [52]. Since no small particles are present, the effect of the in-line atmospheric plasma treatment leads to enhanced adhesion between the PP and the PA6. Additionally, the shape of the dispersed PA6 may influence the impact resistance as mentioned in [52].

polypropylene-polyamide 6-blend untreated

polypropylene-polyamide 6-blend treated with atmospheric plasma

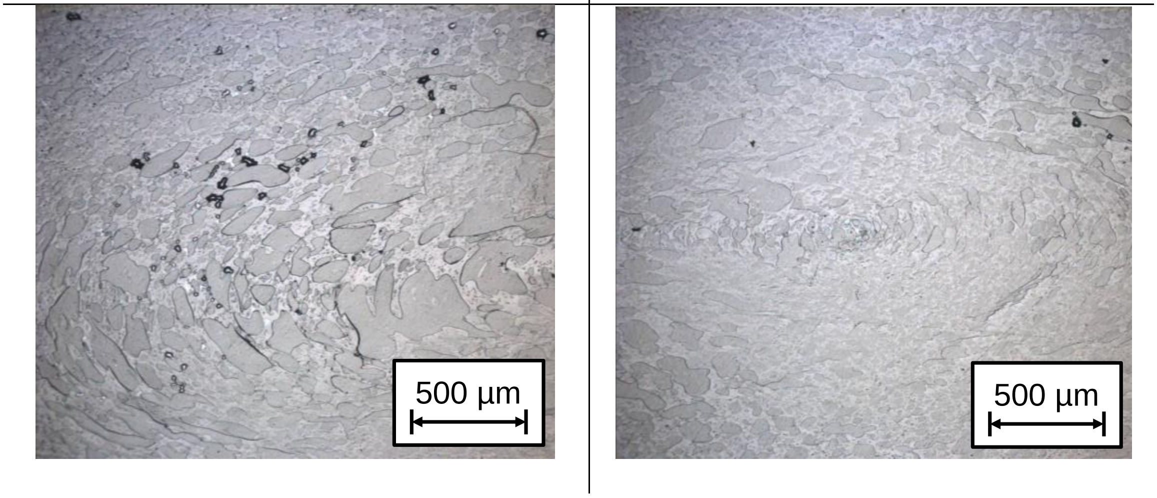

Figure 2: Comparison of the morphology of untreated and treated PP-PA6-blend by using an optical microscopy.

4 CONCLUSIONS AND PERSPECTIVE

This work introduces a novel approach for an in-line atmospheric plasma-treatment process during extrusion. The application of the plasma-treatment leads to a slight increase of the tensile properties, of both, short glass fibre reinforced PP and PP-PA6-blend. But the notched impact strength of the blend can be increased significantly, which can be attributed to an improved dispersion of the PA6 as well as enhanced miscibility and adhesion between the PP and the PA6. This shows the enormous potential of the in-line atmospheric plasma-treatment. However, the effects have to be investigated in more detail in further studies, which should include a deeper insight into the material properties as well as into the processing conditions, providing further information about the mechanisms at the interface during the process and the effects of the plasma parameters as well as processing conditions, such as the actually treated surface during processing and the residence time of the materials in the plasma zone.

Apart from an in-depth study of the setup and the systems mentioned in this study, one can think of various other possible setups, such as an in-line plasma coating process using suitable monomers or the incorporation of micro particles to enhance different composite properties. Next to plasma jets other plasma excitation types like dielectric barrier discharge (DBD) are worth to be researched. Furthermore, the in-line plasma-treatment process is not limited to co-rotating intermeshing twin screw extruders. It can be implemented in every process, in which a zone, which is only partially filled with polymer, is realized. Thus, this in-line plasma-treatment process features a high potential for further scientific research and industrial applications.

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