

Influence of the Water Content in Polyamide 6 on Adhesion and Surface Pre-treatment for Adhesive Bonding

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Abstract

To quantify the influence of absorbed water in PA6 on the pre-treatment and bonding process, an unfilled and unreinforced PA6 material is investigated in a dried and saturated state. The material is pre-treated by atmospheric pressure plasma jet (APPJ) with air as process gas and varying jet distances. The surfaces are investigated by contact angle measurements, DSC and FTIR to detect molecular and morphological changes in the surface. To evaluate the bonding strength, samples are bonded with a two-component polyurethane adhesive and a two-component acrylate adhesive and tested in a lap shear test and a tensile test. The results show that water content has a significant influence on the effectivity of the pre-treatment process and the resulting bonding strength and failure mechanism. The adhesion is majorly affected, however these effects do not affect the macroscopic wetting behavior and cannot be measured in contact angles. FTIR spectra and DSC scans do not show significant changes in molecular groups or crystallinity that would explain the observed adhesion improvement in dried samples. High bonding strength is only achieved with adherents at low water content.

Introduction

Polyamides have a relatively low surface energy of approx. 40 mJ/m^2 [1] and correspondingly poor adhesion properties. Pre-treatment of the polyamide surface is therefore necessary for a strong and secure bond. There are various options for pre-treatment, such as mechanical pre-treatment by grinding or blasting, chemical processes such as the use of primers, or physical processes such as plasma or laser pre-treatment. Mechanical processes are mainly used to increase the surface area and to create binding partners through chain breaks [2]. The effectiveness of this type of pretreatment is limited, especially for thermoplastic materials with difficult wetting behavior. The use of primers can lead to a significant increase in bond strength [3]. However, primer application is a critical process that is difficult to automate and involves the use of chemicals that are critical to health and the environment. Physical processes such as atmospheric pressure plasma are less harmful. These pre-treatments do not require any critical chemicals, are easy to automate and at the same time are able to create high adhesive strengths by incorporating functional groups into the polymer surface [3]. Plasma processes are therefore preferable to chemical activation using primers [4].

Atmospheric pressure plasma jet pre-treatment (APPJ pre-treatment) is a widely used industrial process for the pre-treatment of thermoplastics due to its comparatively low system costs, fast process times and good automation capacity [3, 5]. In contrast to corona pre-treatment, individually shaped surfaces can be treated [6] and there are no requirements for electrical conductivity [7]. APPJ pre-treatment often uses a so-called cold plasma with relatively low temperatures, which prevents thermal damage to the material. According to Habenicht, the temperatures of the plasma current are between 50°C and 150°C [1], but higher temperatures of over $1,000^\circ\text{C}$ are also common [5]. The plasma generated by an electrical discharge contains ions, electrons and radicals and is therefore highly reactive. In APPJ processes, the plasma is applied to the surface by a gas flow, where it reacts and activates the surface. At the same time, minor contaminants can be removed due to the physical fine cleaning effect of the plasma [8]. By

varying the process gas, different plasmas and correspondingly different functionalizations can be generated [9]. Common process gases include compressed air, oxygen, nitrogen [3], argon [10] or helium-based gas mixtures [11]. The aim is to activate the surface through oxygen or nitrogen functionalization, whereby both mechanisms can occur when compressed air is used as the process gas. Aside from the process gas and system-related parameters such as the shape of the plasma jet, the jet distance to the substrate and the jet speed have the greatest influence on the process result [12, 7]. Particularly with high-energy plasmas, excessive energy input during the pretreatment process can lead to overtreatment. The thermal input or radiation produces oxidized material with a low molecular weight (LMWOM - low molecular weight oxidized materials), so-called molecular scrap on the surface. These LMWOM do not have a strong bond to the substrate and therefore hinder secure bonding. The formation of such groups must be prevented accordingly [13, 14].

While the APPJ pre-treatment for polyamides has been researched in various publications, but the water content in the material has not been thoroughly addressed yet. In a series of publications, Gao et al. investigated the APPJ pretreatment of polyamide 6 films with a gas mixture of helium and oxygen. In T-peel tests, an increase in peel resistance of over 30% was observed due to an increase in the number of oxide groups [11]. Gao et al. also looked at the influence of relative humidity on the pre-treatment process. The humidity of the air had an influence on bond formation. At higher humidity, the number of oxide bonds increased. However, the resulting improvement in peel strength compared to a dry environment was only marginal at less than 10% [15]. In another publication, Gao et al. investigated the influence of the water content in the polyamide on bond formation by APPJ pretreatment. At high water content, a significantly lower chemical change was observed in the polymer than at low water content. Fewer oxide and nitrogen bonds were detected on the surface [16]. These measurements were not validated with mechanical tests in order to make reliable statements about the influence of water content on the adhesion conditions.

Schäfer et al. investigated the surface pretreatment of carbon fiber-reinforced polyamide 6 using APPJ to produce semi-structural bonds [13]. Two unspecified PUR adhesives were used. For the tests, the polyamide 6 material was conditioned to a water content of 0.85% in accordance to EN ISO 1110. The jet distance, jet speed and number of passes were varied. An increase in surface energy and an increase in oxide groups was measured as a result of the pre-treatment. At low jet spacing and speed, the adhesive strength was greatly reduced due to overtreatment. The high thermal energy input resulted in LMWOMs on the surface. The best results were obtained by repeating passes at high speed and jet spacing, i.e. low energy input. Schäfer et al. achieved cohesive fractures at up to 20 MPa in tensile shear tests according to EN 1465, which corresponded to more than a doubling of the initial strength. The effectiveness of repeated passes with low energy input was partly justified by the possible removal of the remaining water content in the surface. However, no further investigations were carried out to investigate this influence [13].

For other joining technologies, the effect of the water content on the respective joining process has been investigated. Götze et al. found that dry polyamide nanofiber materials widen the process window of

laser structuring or laser cutting processes [17]. Van Aaken investigated the influence of moisture in polyamide 6 and polyamide 66 on the ultrasonic welding process. A general influence of moisture on the weld seam quality and the required process time was identified [18]. The laser welding process of polyamides is also significantly influenced by the water content in the material. The water content has an influence both on the absorption behavior of the laser radiation [19] and on the seam quality [20].

The research shows a large knowledge gap in adhesive bonding technology with regard to the effects of the water content in polyamides on the APPJ pre-treatment and properties of the resulting bond. Despite the known significance of the water content and its influence on the material's properties, as well as the knowledge about the influence of other joining technologies, for the APPJ pre-treatment process the exact influence of water has not been understood yet.

Materials and Methodology

Materials

The tests are conducted on an unreinforced PA6 material produced and supplied by Envalior Deutschland GmbH from Dormagen, Germany. The used PA6 is a mostly virgin material with little additives, the properties are summarized in Table 1. The samples are produced by injection molding small sheets which are subsequently cut into the required shapes using a table saw.

Table 1
Overview of investigated PA6 material [21]

Brand name	Color	Water saturation	Flexural modulus
Durethan	Ivory	3% (23°C/50% r.h.)	850 MPa
B30S 000000		10% in water (23°C)	(conditioned)

To separate the influence of possible reactions of components of the adhesive with water, two adhesives of different chemical bases are chosen for the mechanical tests. A two-component polyurethane (PUR) and a two-component methyl-methacrylate (MMA), supplied by 3M Deutschland GmbH from Neuss, Germany are investigated. The adhesives are compared with relevant features in Table 2. While PUR is advertised as a multipurpose adhesive for plastics and metals, MMA was especially designed for bonding polyamides without pre-treatment [22,23]. PUR uses 4,4'-methylenediphenyldiisocyanate oligomers as a hardener, which as an isocyanate will react with water if present. Both adhesives are cured at 60°C for two hours and subsequently stored at room temperature for 22 hours before testing.

Table 2
Overview of investigated adhesives [22,23]

Notation	Brand name	Chemical base	Tensile Strength	Tensile Modulus
			ASTM D638	ASTM D638
PUR	Scotch-Weld DP 6330NS	Polyurethane	20 MPa	980 MPa
MMA	Scotch-Weld DP 8910NS	Methyl-methacrylate	16.7 MPa	785.4 MPa

Surface pre-treatment

The material is cleaned with iso-propyl alcohol for the reference surface (Ref), as well as before the surface pre-treatment. For the pre-treatment, the APPJ system T-SPOT S1 by Tigres GmbH from Marschacht, Germany, is used. The plasma generator provides a power of 1.000 W. The jet is a spot jet with a diameter of 8 mm, compressed air is used as process gas. The jet velocity is set to 100 mm/s, pre-treatment areas are traversed with 8 mm distance between the individual lines for a homogenous pre-treatment. The jet distance to the surface is varied, the investigated distances are 15 mm, 20 mm, and 25 mm. Because the activation by APPJ is time sensitive and the effect dissipates with time, the subsequent bonding and surface analytical measurements are conducted within 20 minutes of treatment.

Testing methodology

For the investigations, two different water contents are set: A dry one ("W0", water content below 0.1 wt.-%) and a saturated one at 23°C and 50% rel. humidity according to the technical data sheet of the PA6 ("W3", 3 wt.-% water content). To precisely control the water content during the investigations, the material is dried in an oven at 60°C for five days. The dried state (W0) is controlled gravimetrically on an analytical scale. For the dried samples, a water content of below 0.1 wt.-% is assumed due to absorbed water from the atmosphere during handling of the samples. Because of long saturation times at air, the W3 samples are adjusted to the saturated state after drying by immersion in purified water at room temperature. The weight of the samples is checked gravimetrically until a total increase of 3 wt.-% of the dry sample weight is met with an error margin of 0.05 wt.-%. After reaching 3 wt.-%, the samples are dried with paper towels and stored in a sealed bag for 24 hours, to level the water content within the sample before further investigations.

The wetting properties of the surfaces is evaluated by contact angle measurement according to EN 828 and calculation of the surface energy with the OWRK model [24, 25]. The measurements are conducted using deionized water, ethylene glycol and diiodomethane p.a. with five measurements per testing liquid. To detect molecular changes due to the plasma activation, the surfaces are analyzed by Fourier-transform infrared (FTIR) spectroscopy. The measurements are conducted with a Nicolet iS20 FTIR Spectrometer by Thermo Fisher Scientific GmbH, Dreieich, Germany, in 16 Scans per measurement on an ATR (attenuated total reflectance) module using a monolithic diamond. A further characterization of the polymer morphology is conducted by differential scanning calorimetry (DSC). Since the plasma process only affects the surface, DSC samples are cut out of the surface using a sliding microtome. Because a to

the knife completely parallel fixture of the sample is impossible at this level, the microtome knife is leveled with the surface in 5 μm steps until it starts cutting into the surface on one side. Then the knife cuts out the sample with a depth of 30 μm to produce a sample with enough mass for DSC. The DSC measurement is conducted using a DSC 300 Caliris Supreme ASC by NETZSCH-Gerätebau GmbH from Selb, Germany. Nitrogen is used as purge gas and the heating rate is set to 10 K/min. Since the changes due to the plasma process are to be characterized, the first heating is analyzed.

To evaluate the effect of the surface pre-treatment mechanically, the bond strength is tested in lap shear tests, as well as in a centrifugal adhesion test (CAT) with both presented adhesives. The lap shear test is conducted according to EN 1465 with 4 mm thick adherents and a bond layer thickness of 0.5 mm for both adhesives. Three tests per parameter are carried out at a testing speed of 2 mm/min. The CAT is carried out in a Adhesion Analyser LUMiFrac by LUM GmbH from Berlin, Germany. Testing stamps with a diameter of 10 mm are used and pre-treated with a laser, to exclude the adhesion to the test stamps as a point of failure in the tests. 40 ml of the adhesives is applied to the test stamps with a pipette, a bonding layer thickness of 0.5 mm is adjusted by using glass pearls to increase reproducibility of the CAT samples. Four samples are tested per at once, for parameters with high deviation due to variances in the samples production the test extent is increased. The failure patterns of the mechanical tests are analyzed according to EN ISO 10365.

Results & Discussion

To quantify the wetting behavior, the polar and dispersive components of the surface free energy is derived from the contact angle measurements. The results are displayed in Fig. 1, divided water content and pre-treatment. The dispersive component does not change significantly between the surface pre-treatments. The polar component increases after pre-treatment with all parameters. 15 mm and 20 mm jet distance lead to highest levels in the polar component. There is no significant difference in the surface free energy between dried and saturated material. Contact angle measurements can therefore not reliably differentiate between a saturated and dried surface, before as well as after pre-treatment.

The FTIR spectra of the different pre-treatment parameters are displayed in Fig. 2. At around 3500 cm^{-1} , the spectra show a minor deviation from the reference surface after all pre-treatment parameters. This area in wavenumber corresponds with the stretching vibration of hydroxyl groups. The observed decrease of absorption after pre-treatment in this area, implies a decrease of hydroxyl groups, which would mean a decrease of functional groups due to the pre-treatment. However, hydroxyl groups are also present in water. Since the observed effect is more distinct in the saturated material (W3) and the pre-treatment parameters show a uniform curve, the observed decrease in hydroxyl groups is interpreted as a surface drying effect of the APPJ pre-treatment. Due to the thermal energy of the plasma, surface-close water is evaporated leading to the observed decrease in hydroxyl groups. The FTIR data does not show a functionalization of the surface by the formation of new functional groups. This observation indicates, that the either FTIR is not the right method to measure the surface functionalization on the investigated

material, or that the mode of action of the applied APPJ process is not the formation of functional groups, but an etch process on (sub-) nanometer scale, as suggested by Arikan et al. [26].

To consider changes in crystallinity due to water content or as an effect of the pre-treatment as a mechanism for improved adhesion, the surface layer is investigated in DSC measurements. The melting peaks of the DSC curves used for calculating the crystallinity are displayed in Fig. 3. The shape of the melting curves does not change significantly, although a minor shoulder prior to the endothermic peak is visible in the curves of the reference samples. This shoulder may indicate an increase of the γ -crystalline phase [27, 28, 29], or a change in crystallite thickness [30]. The crystallinity derived from the DSC data is compared in Table 3. The changes in crystallinity are not significant enough to indicate an influence on adhesion.

Table 3
Crystallinity of the PA6 with varying water content and surface pre-treatment

Water content	Pre-treatment	Crystallinity	Melting peak
0 wt.-%	none	35,19%	222°C
0 wt.-%	25 mm	36,44%	223°C
3 wt.-%	none	35,74%	223,1°C
3 wt.-%	25 mm	34,70%	225,1°C

The data from the mechanical tests with the PUR adhesive is displayed in Fig. 4. The failure patterns are specified in the respective graphs. Because the investigated material was not fiber reinforced, partial substrate failure occurred at low lap shear strength already, due to bending of the samples. This effect is also limiting the maximum lap shear strength for this material at around 10 MPa. In the lap shear test, the saturated reference only reached half the lap shear strength of the dried reference. With the pre-treatment, the lap shear strength was improved for all jet distances and water contents. A jet distance of 20 mm and 25 mm led to highest lap shear strengths. At 25 mm jet distance, there was no adhesive failure visible in the fracture pattern, indicating best adhesion with this pre-treatment parameter. The difference between the dried and saturated states is distinctive, for all parameters the dried state achieved around double the lap shear strength of the saturated state.

The CAT data shows a different progression. There is no significant difference in tensile strength between dried and saturated state for the reference surface. At 20 mm jet distance, the difference in tensile strength with the water contents is already significant. At 25 mm jet distance the dried state leads to double the tensile strength of the saturated state at over 21 MPa. While the saturated material shows a combination of adhesive and cohesive failure, the dried samples fail cohesively and in the substrate. The cohesive failure of the adhesive in the saturated samples at low strengths can be attributed to the reaction of the isocyanates in the adhesive with water. The reaction produces CO_2 which causes surface-close defects in the adhesive. Furthermore, the ratio of components is affected by isocyanates reacting

with water, leading to fewer cross-linking and a weaker boundary layer. The fracture pattern of a lap shear sample of a saturated sample pre-treated at 25 mm depicts this behavior in Fig. 5.

For the isocyanate-free MMA adhesive, the mechanical results are displayed in Fig. 6. In the lap shear tests, the difference between dried and saturated is evident. The pre-treatment improves the lap shear strength at all parameters, with 25 mm jet distance leading to best results for both water content levels. The lap shear strength for the dried material is with 10 MPa at a similar level to the PUR adhesive, at the adherent's strength limit. The failure patterns are a mix of substrate, cohesive and adhesive failure. The tensile strength's progression is similar to the PUR adhesive. While the tensile strength is on the same level at the reference surface, the tensile strength of the dried material increases especially at 25 mm jet distance. The highest tensile strength at 12 MPa is significantly lower for the MMA adhesive compared to the PUR adhesive. For the saturated material, the tensile strength decreases minorly, but not significantly with the pre-treatment.

Conclusions

Absorbed water does not only affect the mechanical properties of PA6, also the adhesion properties are significantly affected by the presence of water. In comparison to dried material, the mechanical tests show a major decrease in lap shear and bonding strength after pre-treatment in saturated material. The comparison of the bonding strength with an MMA and a PUR adhesive illustrates that the observed influence is not solely due to a reaction of the adhesive's components with the water, but an affect on the adhesion properties of the surface. The presence of water suppresses adhesion improvements achieved by the pre-treatment on dried material. However, this difference does not reflect in the macroscopic wetting properties, the surface energy does not change between saturated and dried material, even after pre-treatment. Contact angle measurement cannot be used to determine the adhesion properties in this case and might lead to wrong conclusions if used as a sole control assessment or mechanism. A significant difference in molecular groups to explain the improved adhesion is not detected, FTIR measurements only detects the presence of water. The crystallinity does also not change significantly. These results indicate a different adhesion improvement mechanism from the often cited forming of polar groups at the surface due to the APPJ pre-treatment. The lack of detected chemical changes in the polymer suggests an etching effect of the plasma, roughening the surface on a nanometer or sub-nanometer level, similar to claims by Arikan et al. [26]. In summary however, knowledge about the water content in PA6 is crucial for adhesive bonding processes with non-moisture-hardening adhesives. For best results, the material must be dried before pre-treatment.

Declarations

Author Contribution

C.B. wrote the main manuscript A.S. and U.R. oversaw the works and manuscript

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Data Availability

Data is provided within the manuscript or supplementary information files

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Figures

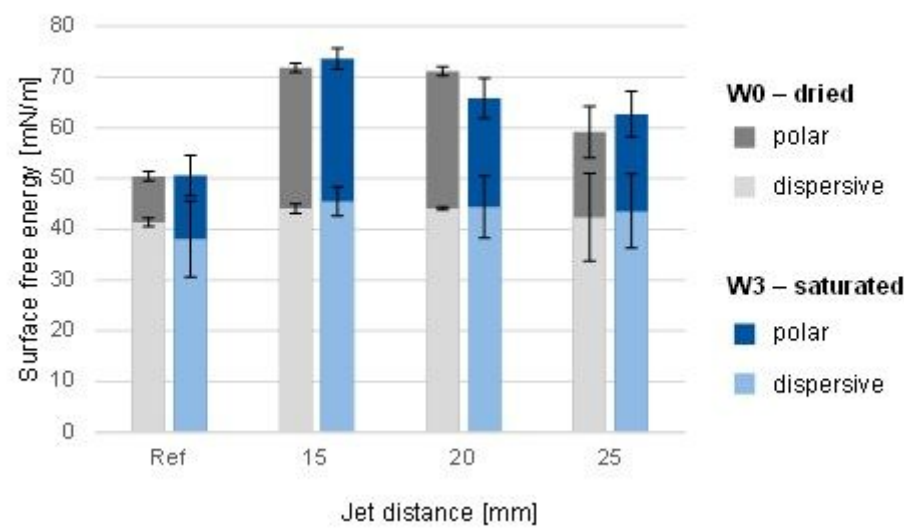


Figure 1

Polar and dispersive components of the surface free energy in dried and saturated state, before and after APPJ pre-treatment

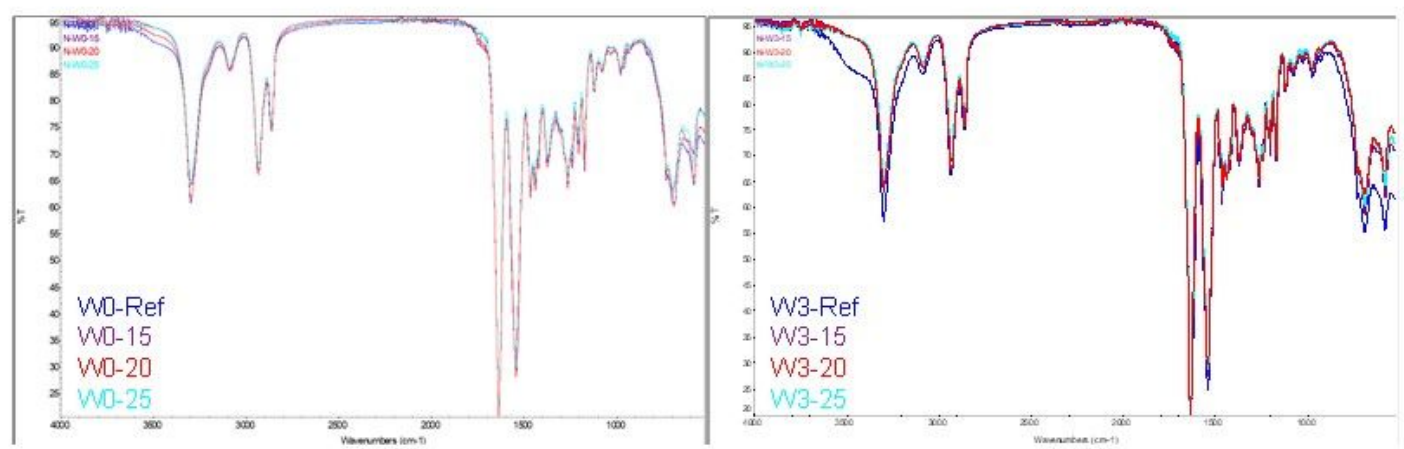


Figure 2

FTIR spectra of PA6 in dried (W0, left) and saturated (W3, right) state with different pre-treatment parameters

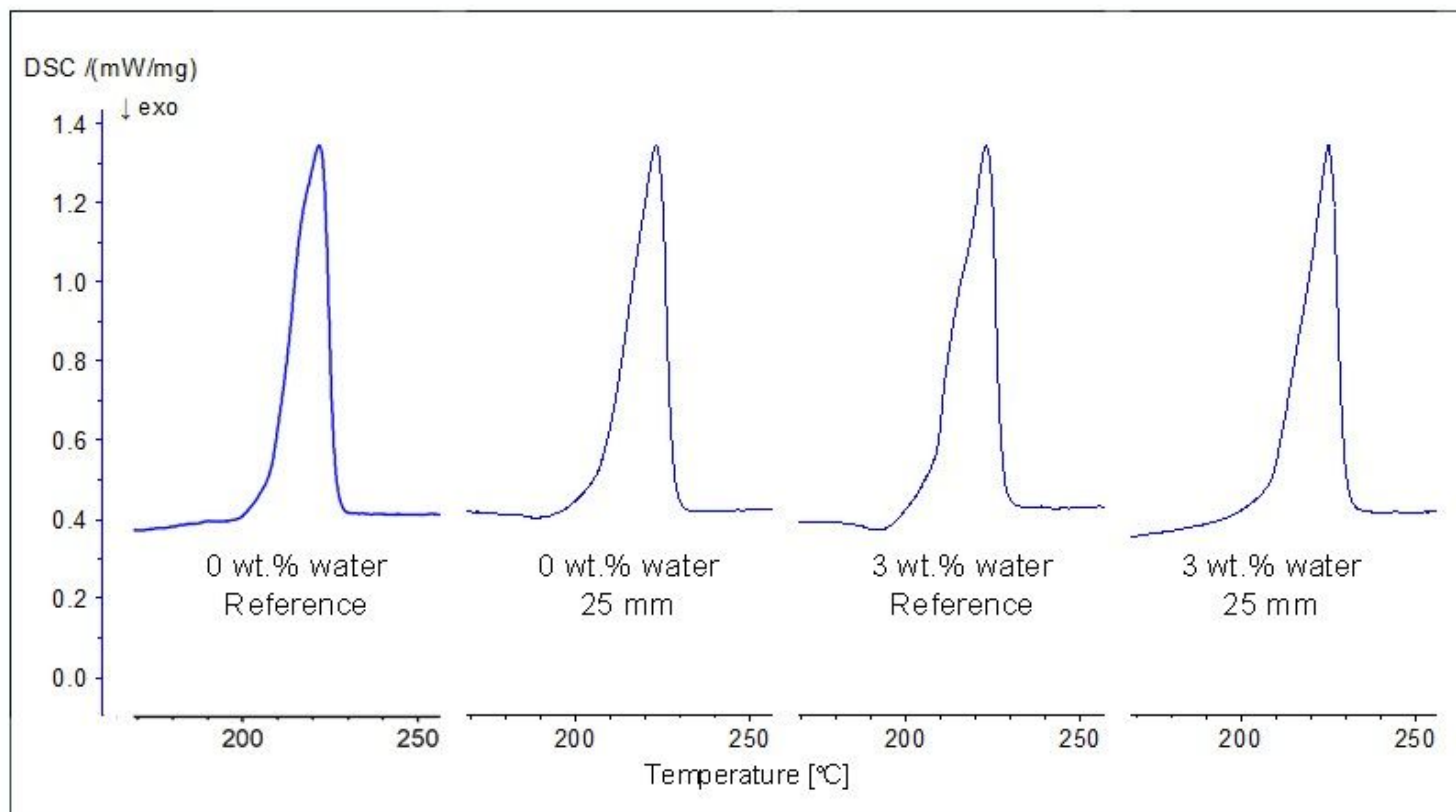


Figure 3

Extract of the melting peaks from DSC scans

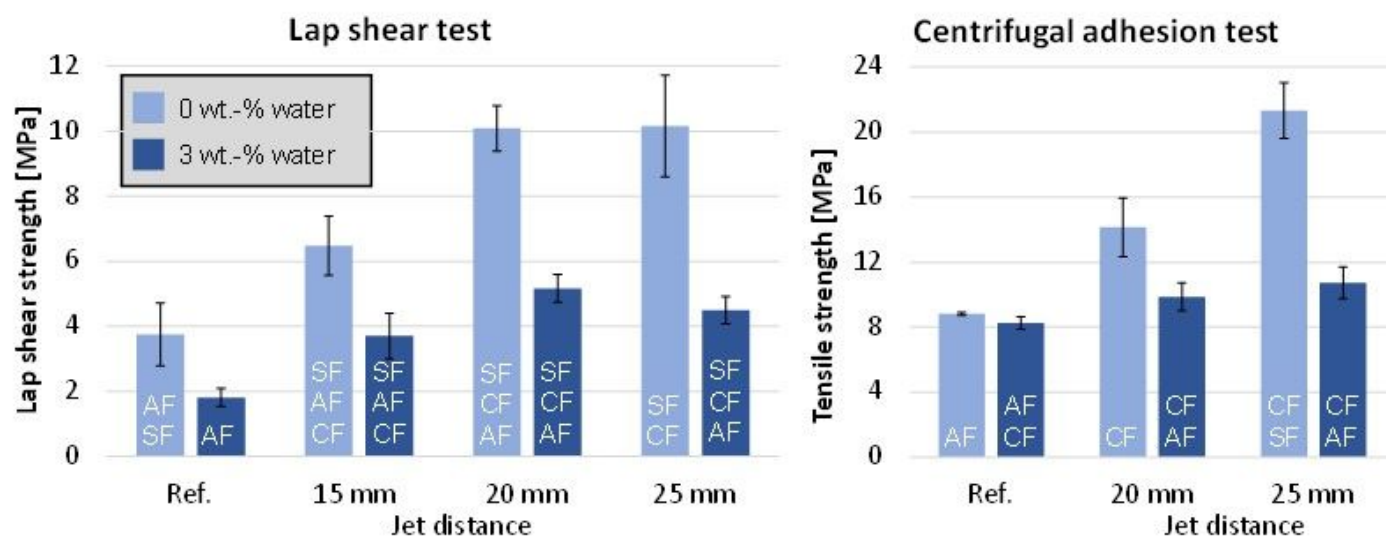


Figure 4

Lap shear strength and tensile strength with the PUR adhesive



Figure 5

Fracture pattern of saturated lap shear sample pre-treated with 25 mm jet distance with extensive defects in the interlayer

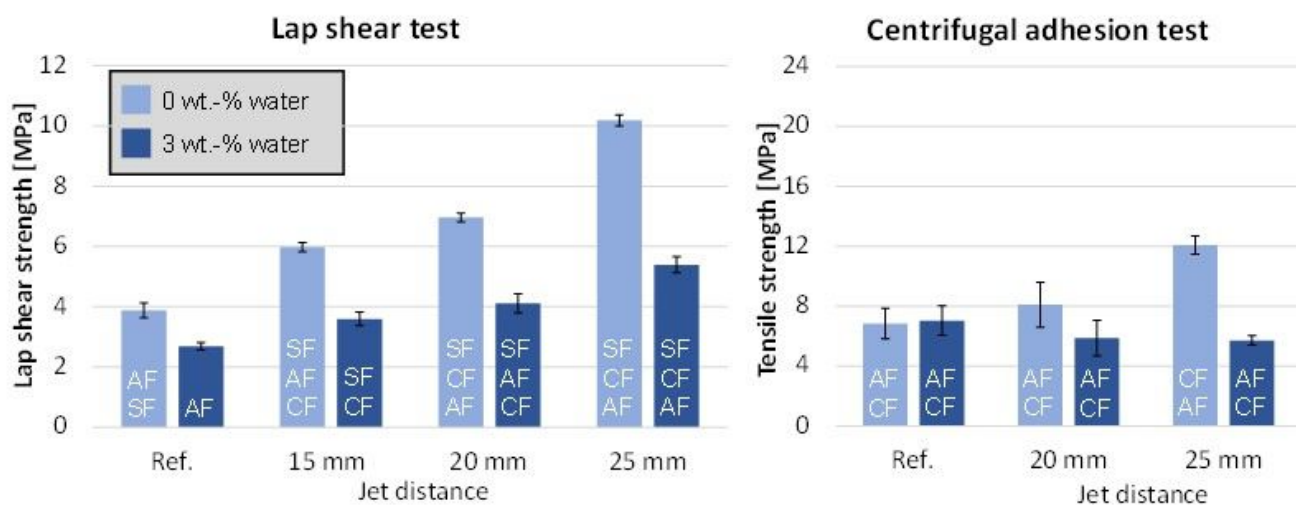


Figure 6

Lap shear strength and tensile strength with the MMA adhesive