PRE-TREATMENT OF CFRP FOR ADHESIVE BONDING USING LOW-PRESSURE BLASTING

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1 Introduction

In the automotive industry the application of composite materials is steadily increasing, due to regulations with regard to the reduction of CO₂ emission and the need to compensate the additional weight of batteries in electric cars. One major challenge limiting the utilization of the full lightweight potential is the joining technology, especially in hybrid structures where composites are bonded to metallic parts. A joining technology with a high potential of taking full advantage of the material is adhesive bonding since it allows for an even stress distribution and does not require holes and thus local reinforcements as conventional joints with fasteners do. However, to achieve a strong and durable bond, surface pre-treatment is often necessary to remove surface contaminations such as residues of release agents.

With the use of FRP in larger quantities it therefore becomes necessary to develop procedures which allow for automated bonding pre-treatment with a high reliability and low process times. As one possible approach to the aforementioned challenge this paper presents low-pressure blasting, a novel blasting process where the grit is not accelerated by positive but by negative pressure inside a closed jet cap and provides results from surface analyses and mechanical testing which have been performed on CFRP surfaces pre-treated by this process.

2 Current industrial solutions for CFRP treatment

As discussed before, surface pre-treatment is a major remaining challenge for the adhesive bonding of composite materials.

In the aerospace industry often peel-plies are used as surface pre-treatment, but these cannot be applied for automotive parts because in the first place the parts have a more complex shape and thus the draping and also the removal of the peel-ply is quite time-consuming. Secondly, the area where adhesive bonding is performed is often close to the exterior shell and thus has to fulfill class-A requirements which are not met by the rough pattern resulting from the removal of peel-ply. This adds to several disadvantages of peel-ply surfaces described in the literature [1-3].

In the last few years, various novel pre-treatment processes were investigated which have a high degree of automation and show a good reproducibility as regards the surface characteristics. Especially the pretreatment by means of laser beam or atmospheric-pressure plasma shall be mentioned and have also been investigated by the authors [4-11]. However, both procedures have the disadvantage of causing comparatively high investment costs. Furthermore, for the laser pretreatment only limited preliminary studies are available. The plasma treatment has the well-known disadvantage that the thickness of contaminated layers which can be removed is limited.

For the reasons named above, especially in the automotive industry the most commonly applied method for surface pre-treatment is manual abrading, which, due to its simplicity, can be done cost-effectively. But the high personnel costs and the limited reproducibility resulting from the mainly manual operation restrict the usability of this procedure in future large-scale manufacturing.

Apart from the simple procedure, main disadvantages of the process are the necessity for prior and subsequent cleaning which requires the application of solvents, the strong dependence of the amount of abrasion on the normal force applied and thus the comparably bad process reliability and the elaborate way of automating the process for complex geometries. Positive pressure-blasting processes are an alternative to mechanical removing of
contaminations but the process generates dust which makes encapsulation necessary and limits the potential of integrating the process into a production line. Furthermore, also subsequent cleaning is necessary to remove residues of grit and abrasion products from the surface.

3 Low-pressure blasting

For the reasons mentioned above, low-pressure blasting was investigated in the work presented here as a possible alternative for the bonding pre-treatment of fiber-reinforced composites, in the light of integrating the procedure into the manufacturing process. The procedure was developed and patented by GP Innovation GmbH, Germany. The investigations were done within a publicly sponsored project. The low-pressure blasting process uses the same mechanical abrasion mechanisms as conventional high-pressure grit blasting, but the grit is accelerated by a negative pressure inside a closed jet cap which is provided by an industrial vacuum cleaner. Figure 1 shows the schematical build-up of the low-pressure blasting jet cap. The jet cap is sealed to the surface by a dynamic gasket which enables lateral movement of the jet cap relative to the surface. Compared to conventional positive pressure grit blasting the process has the advantages that firstly, due to the encapsulation the surroundings are not contaminated with dust, and secondly the grit and abrasion products are directly removed by the flow going to the suction device. For this reason, a post-treatment of the treated surfaces - unfavorable from the process perspective - is not necessary. Apart from the advantages mentioned above, the procedure is characterized by low investment costs, compared to laser or plasma plants. In addition, the process can be well automated, because the jet cap can be mounted on a robot, as, for instance, used for mechanical trimming of the edges of RTM components. This way, the reproducibility of the operating process is also guaranteed.

The ablation of surface layers, e.g. release agent residues, necessary for an effective pre-treatment, is caused by an erosion process on the surface, as in the case of high-pressure blasting or grinding [3]. When the grit hits the surface, the kinetic energy is used to detach particles from the surface layer whereby a local ablation occurs. This ablation is significantly determined by the blasting material used, i.e. particle size and material, as well as the speed of the particles which again depends to a great extent on the pressure difference between cap and atmosphere. Apart from the kinetic energy of the individual particles resulting from these parameters, the resulting surface also depends on the processing time, i.e. the speed with which the jet cap is moved across the surface, and the mass flow of the blasting material.

4 Materials and Methods

The pre-treatment was performed with a laboratory size low-pressure blasting device provided by GP Innovation GmbH, Germany. The jet cap was mounted to a fixed rack; the specimens were moved underneath the rack by a two axis industrial x-y table. The feed rate was controlled by the speed of the x-y table.

In the first step, with the aim of gathering more information about the process a high-speed video camera was used to determine the speed of the grit particles when leaving the inlet tube. For this purpose a special jet cap with a glass window was used.

This paper shows results which were gathered using four different kinds of grit particles, one was finely ground Na₂CO₃, the three others were broken glass particles with an average grit size of 180, 500 and 1000 μm.

The lap shear and peel tests described in this work have been performed on two different material combinations representing a typical automotive and aerospace configuration of adherends and adhesive.

Table 1: Material configurations

<table>
<thead>
<tr>
<th>Automotive configuration</th>
<th>Adherent 1</th>
<th>Adherent 2</th>
<th>Adhesive</th>
</tr>
</thead>
<tbody>
<tr>
<td>RTM CFRP EP matrix</td>
<td>Aluminum, electro-coated</td>
<td>2 part epoxy</td>
<td></td>
</tr>
<tr>
<td>Aerospace configuration</td>
<td>Prepreg UD CFRP EP matrix</td>
<td>Prepreg UD CFRP EP matrix</td>
<td>1 part epoxy film</td>
</tr>
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</table>

In addition to the mechanical tests surface analyses have been performed by optical light and scanning electron microscopy. Furthermore, a rough
estimation of the surface wetting has been performed by the use of contact angle measurements.

Within the framework of the experiments described in this paper the kind of grit, the size of the particles, the pressure difference between jet cap and atmosphere and the treatment speed were varied. To achieve comparable results in all cases the mass flow of the grit was held constant at 300 g/min.

5 Results

5.1 Characterization of the blasting process

In the first step of the experiments the kinetic energy of the particles was calculated, based on the measured velocity and the average particle size and their mass. The particle velocity was measured by using a high-speed video camera shooting 11200 frames per second. Particle tracking and calibration to a defined length in the picture were used to determine the average speed of the particles. Figure 2 shows a picture series which was used to measure the particle velocity – the average time between two of the pictures is 89 µs. Figure 3 shows the dependency of the particle velocity on the nominal diameter and pressure difference between jet cap and atmosphere.

To determine the average diameter and the size distribution of the different blasting granulates a vibration sieve tower was used. The size distribution was afterwards specified by weighing the mass of granulate in the different sieves and putting it into relation to the total weight.

To ease the particle tracking these experiments were carried out using a reduced mass flow of 50 g/min – comparative analyses of a mass flow of 300 g/min used for the specimen treatment showed that the influence of the mass flow on the particle speed is less than 8 % and thus negligible compared to the spread caused by particle size fluctuation.

To determine the average diameter and the size distribution of the different blasting granulates a vibration sieve tower was used. The size distribution was afterwards specified by weighing the mass of granulate in the different sieves and putting it into relation to the total weight. The average weight of a single particle cannot be determined by weighing because of the very low particle size and thus the comparably high spread caused by inaccuracy of the weighing unit. For this reason the average particle weight was calculated by multiplying the average grain volume with the density of glass (2.5 g/cm³). To validate this assumption of the density, 100 glass particles were weighed and a density of 2.28 g/cm³ was calculated. Taking into consideration that the particles were idealized as spheres and the 100 grains were taken statistically, the error of less than 10 % is acceptable. Based on this assumption the average weight of the particles was calculated as shown in Table 2.

The combination of all the information described above enables the calculation of the kinetic energy and setting it into relation to the particle size of the broken glass particles and the difference between atmospheric and jet cap pressure. This information is given in Figure 4.

Table 2: Calculated average particle mass

<table>
<thead>
<tr>
<th>Granulate</th>
<th>Avg. diameter [µm]</th>
<th>Weight [µg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂CO₃</td>
<td>155</td>
<td>4.9</td>
</tr>
<tr>
<td>Broken glass GB 180</td>
<td>146</td>
<td>3.8</td>
</tr>
<tr>
<td>Broken glass GB 500</td>
<td>374</td>
<td>68.5</td>
</tr>
<tr>
<td>Broken glass GB 1000</td>
<td>765</td>
<td>586</td>
</tr>
</tbody>
</table>

5.2 Characterization of treated surfaces

Surface characterization has been performed by roughness measurements, optical microscopy, scanning electron microscopy (SEM) and by characterization of the surface energy using contact angle measurements.

The surface roughness measurements have been performed using a confocal laser scanning microscope. For these measurements, feed rate, jet cap pressure and nominal grain size were varied. Table 3 sums up the measured average roughness Rₐ and depth of roughness Rₜ.

Table 3: Surface Roughness

<table>
<thead>
<tr>
<th></th>
<th>Rₐ</th>
<th>Rₜ</th>
</tr>
</thead>
<tbody>
<tr>
<td>untreated</td>
<td>0.142</td>
<td>2.82</td>
</tr>
<tr>
<td>Varied feed rate; GB180; -200 mbar jet cap pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>840 mm/min</td>
<td>2.007</td>
<td>11.044</td>
</tr>
<tr>
<td>2400 mm/min</td>
<td>1.723</td>
<td>14.436</td>
</tr>
<tr>
<td>6000 mm/min</td>
<td>0.652</td>
<td>7.287</td>
</tr>
<tr>
<td>Varied jet cap pressure; GB180; 2400 mm/min</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-200 mbar</td>
<td>1.723</td>
<td>14.436</td>
</tr>
<tr>
<td>-150 mbar</td>
<td>0.951</td>
<td>6.682</td>
</tr>
</tbody>
</table>
These results demonstrate increasing roughness and thus treatment intensity if either the feed rate is reduced, or the jet cap pressure or nominal particle size are increased. However, the use of larger particles leads to a more inhomogeneous treatment of the surface because the mass flow was kept constant and thus fewer particles hit the surface. The correlation between kinetic energy of the particles and the achieved average roughness is illustrated in Figure 5.

Figure 6 shows images taken by optical microscopy from three differently treated surfaces. Picture a) shows the surface treated with the lowest intensity, it is clearly visible that some parts of the surface have been treated and that the surface is slightly structured. Picture b) shows an increased treating intensity compared to a). Resin is removed and fibers are getting locally exposed. Further increase of the treatment intensity creates a surface as shown in c), fibers are exposed and the top resin layer is removed completely. No particles or abrasion products are visible on any of the surfaces investigated.

SEM pictures of CFRP surfaces blasted with broken glass particles of a nominal size of 180 µm, a jet cap pressure of 200 mbar and different feed rates as well as untreated and grinded references are shown in Figure 7. The untreated surface shows some dust and contamination and also a very smooth surface correlating with the low roughness values presented in Table 3. The surfaces which were abraded with P80 sanding paper show deep scratches and destroyed fibers but also areas that appear to be as the untreated surfaces and may thus not have been treated sufficiently. As already described in the optical microscope pictures in Figure 6 and correlated with the roughness values in Table 3, the intensity of abrasion increases if the feed rate is reduced. The specimen which is blasted with a feed rate of 840 mm/min shows clearly exposed fibers, the one treated with 6000 mm/min only locally exposed fibers and some craters caused by the impact of single particles, for this feed rate no laminar treatment of the surface is achieved.

Figure 8 shows a comparison of the surface energy before and after low-pressure blasting with glass and \( \text{Na}_2\text{CO}_3 \) particles. For this test it has to be taken into consideration that the change in surface roughness also influences the wetting angle of the test liquids. However, there is a significant increase in wettability for both kinds of particles, which is presumably caused by the removal of release agent residues by the mechanical abrasion. Furthermore the increase of the polar component of the surface energy due to the \( \text{Na}_2\text{CO}_3 \) particle blasting is significant. This might be caused by chemical interaction between the \( \text{Na}_2\text{CO}_3 \) and the epoxy resin which is possible, due to the agile electrons inside the \( \text{Na}_2\text{CO}_3 \) molecules.

### 5.3 Mechanical tests

In the course of the testing program described in this paper two different material configurations were investigated as described in Table 1. For these two combinations different failure modes can occur. These are presented in Figure 9. For the aerospace configuration either adhesion failure, which means interfacial failure between the adhesive and the adherent, or cohesion failure inside the adhesive was observed. These are well known for adhesive bonds and will be tagged AF or respectively CF in the following. The automotive material configuration also showed failure inside the top layer of the CFRP adherent and failure between the aluminum adherent and the e-coating that was applied to the aluminum in a serial production process. These failure modes will be tagged as CSF (cohesive substrate failure) and e-coat. The latter two failure modes indicate that the pre-treatment which was in the focus of the work presented here allows the full utilization of the strength of the adherends. However, to achieve higher overall strengths an optimization of the e-coating and the automotive CFRP should be investigated.

The results of the single lap shear tests which show the above-mentioned failure modes are presented in Figure 10. The specimens manufactured from the aerospace setup show complete adhesion failure if no pre-treatment is applied. This is caused by the high contamination of the surface with residues of release agents already described by the authors on

<table>
<thead>
<tr>
<th>Pressure</th>
<th>Feed Rate</th>
<th>Roughness Values</th>
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<tbody>
<tr>
<td>-105 mbar</td>
<td>0.914</td>
<td>3.972</td>
</tr>
<tr>
<td>Varied nominal particle size; -150 mbar; 2400 mm/min</td>
<td></td>
<td></td>
</tr>
<tr>
<td>GB180</td>
<td>0.951</td>
<td>6.682</td>
</tr>
<tr>
<td>GB500</td>
<td>2.196</td>
<td>11.615</td>
</tr>
<tr>
<td>GB1000</td>
<td>3.890</td>
<td>11.245</td>
</tr>
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</table>
the identical material in [13]. The low-pressure blasted specimens fail completely cohesively if treated with the lowest feed rate of 840 mm/min. Increase of the feed rate to 2400 mm/min leads to an amount of approx. 10 % adhesion failure and decrease of the lap shear strength of about 3.5 MPa. Further increase of the feed rate causes complete adhesion failure and a significant reduction in bond strength of about 11 MPa compared to the specimens treated with an 840 mm/min feed rate. This may be explained by the decreasing intensity of the blasting process which was shown in the SEM surface images presented in Figure 7.

The specimens manufactured from the automotive material combination also show complete adhesion failure if the surface is not pre-treated before adhesive bonding. However, all of the specimens that were pre-treated by low-pressure blasting regardless of the feed rate show either failure inside the e-coating or inside the CFRP adherend. Compared to the aerospace setup the average lap shear strength is significantly lower for all feed rates and under consideration of the spread there is no decrease of the bond strength with increasing feed rate. This is caused because the e-coat and the RTM CFRP itself have a comparably low cohesive strength and thus the failure starts inside the adherends if a threshold value of the adhesion strength is achieved by the pre-treatment. Looking at the aerospace specimens treated with 6000 mm/min, which show adhesion failure at a higher strength than all of the automotive type specimens, it becomes plausible that this threshold is already achieved at the fastest treatment speed. Thus in the automotive material combination the adherents limit the maximum bond strength.

To visualize the results and to take a look at a different kind of loading that might be more sensitive to surface effects, roller peel tests have been performed for the aerospace material combination. For this purpose aluminum sheets with a thickness of 0.5 mm have been sandblasted and cleaned and were afterwards bonded to the aerospace CFRP. The CFRP sample was not completely pre-treated with one parameter, but three approx. 25 mm wide areas have been treated with three different feed rates on each sample. Figure 11 vividly illustrates the correlation between failure mode and peel load for the three different blasting parameters or feed rates. With an increased feed rate the portion of adhesion failure increases and in the areas with this failure mode the load significantly decreases. In the spaces between the pre-treated zones complete adhesion failure occurs at about five times lower loads.

With the aim to get a first impression of the ageing behavior of bonds pre-treated by low-pressure blasting aerospace setup specimens were pre-treated with 840 mm/min feed rate using Na₂CO₃ and GB180 particles. The lap-shear specimens were afterwards aged for 2000 h at 70 °C in a water saturated atmosphere.

The results of the lap shear tests after ageing are shown in Figure 12. After ageing the failure mode of the specimens treated with broken glass particles moves slightly into the adherents but stays mainly cohesive inside the adhesive. This is caused by the moisture induced reduction of the cohesive strength of the adhesive and matrix resin. However no interfacial failure appears which means that the pre-treatment also works well after ageing, which again is an indicator that the release agent residues are completely removed from the surface. In contrast the specimens which were low-pressure blasted using Na₂CO₃ change their failure mode to complete adhesion failure after ageing. This correlates with the idea of activation due to agile electrons. In this case only surface activation is achieved but the release agent is not removed thoroughly.

6 Conclusions

The increasing application of composite materials in the automotive as well as in the aerospace industry leads to the demand for a cost-efficient, well automatable and process-stable pre-treatment process for adhesive bonding. The investigations presented in this paper show low-pressure blasting as a possible alternative to conventionally applied processes like manual grinding or high-pressure grit blasting. The applicability of the process as well as the influence of several process parameters were investigated for an automotive and an aerospace material combination.

It has been shown that, by application of this technique, a change of the failure mode from adhesion failure on untreated surfaces to either complete cohesion failure inside the adhesive or failure of the adherents is achievable. In the case of the aerospace setup this involves an increase of the
lap shear strength by a factor of nearly 13. For the automotive setup used in these investigations the increase is about 1.7 times higher despite the low strength of the adherents.

The process parameter with the highest impact on the pre-treatment result was found to be the feed rate. The parameters described here achieve an area rate of about 1 m²/h. Further investigations will focus on the potential of increasing this area rate by a combined increase of feed rate and particle mass flow.

7 Acknowledgements

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The low-pressure blasting technology has been patented by the GP Innovation GmbH, Lübbenau, Germany.

References

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PRE-TREATMENT OF CFRP FOR ADHESIVE BONDING USING LOW-PRESSURE BLASTING

Fig. 1: Schematic design of a low-pressure blasting device

Fig 2: Picture series showing a particle moving downwards

Fig. 3: Dependency of grain velocity on jet cap pressure and nominal grain size

Fig. 4: Dependency of the kinetic energy of the particles on the grit size and jet cap pressure

Fig. 5: Correlation between average roughness and kinetic energy

Fig. 8: Polar and disperse parts of the surface energy measured by contact angle measurements.
Fig. 10: Lap shear strength of specimens pre-treated by low-pressure blasting

Fig. 12: Lap shear strength before and after ageing; specimens treated at 840 mm/min feed rate

Fig. 6: Optical microscope images of CFRP surfaces treated by low-pressure blasting

Fig. 7: SEM pictures of differently treated CFRP surfaces
Fig. 9: Failure modes for the aerospace and automotive configuration

Fig. 11: Load displacement curve correlated with fracture surface after roller peel test