Improving the Accuracy of Deep Drawn Fiber-Metal Laminate Parts by Preliminary Surface Treatment

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Abstract. In the present study, fiber-metal laminates (FMLs) were formed by deep drawing after the metallic component was pretreated by laser structuring, sandblasting or anodizing in order to reduce the height of process-related wrinkles. Such surface texturing is commonly performed to improve the bond strength between the adhesively joined components of the FML by selectively increasing the surface area. This results in improved stability of the finished FML part due to increased resistance to delamination. First, the adhesion properties induced by each process were quantified using shear tensile tests. The shear strength was increased for all processes compared to the unstructured surface. The surface structures were characterized by roughness measurements and scanning electron micrographs. The surface properties adjusted by the different processes also contributed to the part accuracy of the deep-drawn FMLs. During forming, the matrix material contained in the unidirectional fiber composite plastic tends to build up against the forming direction, resulting in wrinkling between the flange and the cup shell. The increased adhesive strength of the metallic component impedes the flow of the matrix material and decreases the height of the resulting wrinkles. The height of the wrinkles was determined using a coordinate measuring machine on the formed FML cups.

1. Introduction
The climate crisis is forcing automobile, aircraft and ship builders to make a major change in the use of materials. Lightweight materials are necessary to reduce energy consumption and thus minimize CO₂ emissions. The use of fiber-metal laminates (FML) is one promising way to meet the stringent requirements, as they combine advantageous properties such as the ductility of steel and the high stiffness of carbon fiber reinforced plastics (CFRP) into high-performance hybrid materials. [1, 2]

The most common way of joining FML components together is adhesive joining. This technique ensures a two-dimensional force transmission as well as the galvanic isolation of the components to avoid contact corrosion. [3, 4] The surfaces should always be sufficiently pretreated beforehand. Prerequisite is a grease-free surface, which can be achieved by simple cleaning, e.g., with acetone. In addition, the surface can be enlarged or roughened to achieve a mechanical bond between the cured adhesive and the substrate. [5] Improved adhesion between fiber composite plastic and metal significantly enhances the strength properties of an FML component [6]. Various methods are available
for this purpose. A very well-known process is sandblasting, in which compressed air is used to blast the abrasive at high speed onto the surface, thus deforming it. The resulting undercuts in the surface structure create spaces into which the adhesive can flow and cure during the joining process. This produces a mechanical bond that ensures good adhesion properties. [7] Another method for surface pretreatment of metals is laser structuring with a pulsed laser source, which has both a cleaning effect and can enlarge the surface microscopically and nanoscopically. While the microscopic surface expansion originates from the craters that form during the resolidification of the material, the nanoscopic surface expansion results from particles that are ejected from the molten pool and then land back on the surface and solidify. [8, 9] Another process is anodizing, an electrolytic pretreatment to create nanoporous oxide layers with excellent wetting properties, making it a very good basis for adhesive joining processes. [10, 11]

It is possible to further process fiber-metal laminates by forming operations such as deep drawing. This opens up a wide range of potential applications, including the production of highly rigid car body components. [4, 12, 13] During forming, the matrix material contained in the unidirectional fiber composite tends to build up against the forming direction, resulting in wrinkles between the flange and the cup shell. [12] The increased bond strength between the metallic component and the fiber composite plastic impedes the flow of the matrix material and could reduce the height of the resulting wrinkles. This would lead to improved manufacturing accuracy of FML components, another positive effect in addition to the good strength properties. The aim of this paper is to investigate the influence of surface structuring of zinc coated steel sheets by means of sandblasting, laser structuring and anodizing on the adhesion properties of the fiber-metal laminate and the resulting manufacturing properties such as the accuracy of deep drawn FML cups. The process chain is presented below.

2. Experimental setup

2.1. Materials

The metallic component subjected to the various surface pretreatments was CR290Y490T-DP-EG [14], a zinc coated dual-phase steel. The material thickness was 0.55 mm. This material is mainly used in the automotive industry due to its excellent lightweight properties. The second component of the FML was a 0.3 mm thick unidirectional carbon fiber reinforced polymer (CFRP) prepreg called SIGRAPREG C U230-0/-NF-E320 [15]. Furthermore, the film adhesive HexBond™ 677 [16], which was specially developed for joining metals with fiber composites, was chosen to produce the shear tensile specimens. No adhesive was used in the combined forming process as the components were intrinsically joined.

2.2. Surface pretreatment

The steel sheets, the metallic component in the FML, were pretreated with three different processes. First, the steel sheets were cut into two different shapes. While 20 sheet metal strips measuring 100 x 22 mm² were cut on a plate shear for the shear tensile tests, 40 circular sheet metal blanks with a diameter of 180 mm were laser cut for the deep drawing tests. The sheets were then pretreated with the appropriate processes, the sheet strips being textured on both sides and the circular blanks on one side.

The sandblasting was carried out with a compressed air unit made by MHG of the SMG 160 S/D type and a corundum with a grain size of F220 as the abrasive. A blasting pressure of 3.5 bar was employed with an angle of 70 - 80° to the surface. The laser structuring was performed with a Q-switched pulsed YVO4 laser (MD-X1520C) and the following parameters: pulse frequency f = 25 kHz, scan speed v = 1500 mm/s, output power Pa = 25 W, line spacing L = 0.08 mm. The direction of the laser patterning on the circular sheet metal blanks is shown in Figure 1. In contrast to laser-structured surfaces, sandblasted and anodized surfaces have direction-independent properties, which is why a schematic representation is not necessary.
Figure 1: Scanning direction of the laser on the circular sheet metal blanks

The anodizing was carried out with a self-developed system for anodizing zinc coated steel sheets up to a size of 640 x 380 mm², which is shown in Figure 2. The tank of the equipment was filled with an electrolyte in which the sheet to be anodized was immersed before the current source was switched on for a defined time and current. A water solution of potassium hydroxide (KOH) was used as the electrolyte for a substance quantity of 1 Mol. The current density was 2.5 A/dm² and the anodizing time 1 min.

Figure 2: Schematic diagram of the anodizing unit for the galvanized steel sheets 1: lead cathode, 2: steel sheet, 3: tank for electrolyte

2.3. Surface characterization
The surface roughness values Ra and Rz of the differently pretreated surfaces were determined with the HOMMEL-ETAMIC T8000 tactile roughness tester, each surface being measured five times. The arithmetic mean was then calculated from the results. Subsequently, the generated surface morphology was examined in a Zeiss Ultra Plus scanning electron microscope (SEM). The images were taken with a secondary electron detector (SE2) at an accelerating voltage of 5 kV and a working distance of 3.6 - 3.7 mm.

2.4. Lap shear tests
For the shear tensile tests, first the double overlapped shear tensile specimens were fabricated. The already cut out sheet metal strips were each cured with two CFRP strips with the adhesive in a suitable
fixture at 150°C and a joining pressure of 350 kPa for 4 min. These were the ideal adhesive curing conditions, as specified by the manufacturer. The overlap length of the joining zone was 6.5 mm. The arrangement of the components shown in Figure 3 reduces bending during the shear tensile tests.

Figure 3: Double overlapped shear tensile specimen 1: CFRP strips, 2: steel strip

Subsequently, the shear tensile tests were performed on the MTS Criterion® electromechanical universal testing machine at a test speed of 1 mm/min. The shear strength was obtained by measuring the maximum force at failure of the bonded joint and dividing it by the bond area. Five shear tensile tests were performed for each surface condition, i.e. anodized, sandblasted, laser textured and as-received.

2.5. Deep drawing of FML

As can be seen in Figure 4, the active die elements for the combined forming and curing process consisted of a die, a blankholder and a punch, into which heating cartridges were inserted. The punch is cylindrical and has a diameter of $d_p = 99$ mm and an edge radius of $r_p = 10$ mm. The cylindrical die has an inner diameter of $d_D = 105$ mm and a drawing edge radius of $r_D = 10$ mm. The drawing depth used in the tests was $h = 37$ mm. In addition, temperature-stable fluoropolymer foils ($s_0 = 12$ µm) were applied, which were inserted between the die elements and the semi-finished product. This film served to prevent contamination of the die elements by escaping epoxy resin and to minimize friction between the tool surface and the FML blank.

Figure 4: Schematic image of the semi-finished product and the combined forming and curing process, 1: blank holder, 2: punch, 3: heating cartridges, 4: die

The die elements were placed in a high-precision column guide frame to keep the blank holder and die ring parallel. Then, the entire assembly was placed in a hydraulic press. After the active die elements were tempered to the curing temperature of $T_c = 140$ °C, the FML blanks were centered within the die.

| 1) Semi-finished Products | 2) Forming and Curing Process | 3) Finished Parts |
|----------------------------|-------------------------------|------------------|
| FML-Sheet                  |                               |                  |
| a) Metal Sheet CR290Y490T-DP-EG | $s_0 = 0.55$ mm |                  |
|                            | $D_0 = 180$ mm                |                  |
| b) CFRP, 1 layer           |                               |                  |
|                            | $s_0 = 0.30$ mm               |                  |
|                            | $D_0 = 180$ mm                |                  |

Curation Time: $t_c = 10$ min
Curation Temperature: $T_c = 140$ °C
assembly and the thermoforming process was started. The blankholder applied normal stresses to the FML blank, thus suppressing wrinkling of the flange area caused by tangential compressive stresses during deep drawing of the blank. The blankholder force $F_{BH,1}$ was 260 kN, the force $F_p$ exerted by the punch was 500 kN. After the forming process, the deep-drawn FML blank remained in the closed mold for a curing time of $t_{c} = 10$ min. These curing conditions for the CFRP prepreg were based on previous studies as they result in optimum curing of the epoxy resin of the matrix material and thus maximum strength of the prepregs.

2.6. Measurement of the wrinkle height

The height $h_{wr}$ of the wrinkles formed during deep drawing above the cup height $z_{md}$ (see Figure 5) was measured with the Zeiss Contura G2 7/7/6 RDS coordinate measuring machine.

![Figure 5: Wrinkle height $z_{md}$ and measuring depth $h_{wr}$](image)

The measurement is carried out over six measuring depths $z_{md}$, in which the probe records 2000 measuring points on a circular path in each case. The measurement process is illustrated in Figure 6, the measuring height $z_{md}$ refers to the flange underside of the cup.

![Figure 6: Measurement of the wrinkle height](image)

3. Results

3.1. Surface morphology

Figure 7 shows the SEM images of the different surface textures, each with two different magnifications. When comparing the anodized surface (Figure 7 b1) with the initial state (Figure 7 a1), only a slight difference can be seen, as the bright elevations visible in the initial state were slightly reduced by the anodizing process. At higher magnification, however, there is no longer any similarity between the surfaces. The zinc flakes shown in Figure 7 a2 have completely transformed into porous zinc oxide. Furthermore, the sandblasted surface (Figure 7 c1 and c2) indicates a very strong plastic deformation, as deep indentations and undercuts are visible. Figure 7 d1 shows the zinc surface melted and resolidified by laser structuring. Higher magnification demonstrates that particles have settled in the craters.
3.2. Surface roughness

Figure 8 shows the surface roughness Ra and Rz as a function of the surface texturing. It can be seen that the highest roughness is achieved by sandblasting, with Ra increasing by approx. 30%, and Rz by 128% compared to the as received state. In contrast, anodizing reduced both values by 37% and 20%, respectively. Laser structuring did not significantly change the Ra value, the Rz value increased by 18%.
3.3. Shear strength
The shear strength was improved by all three surface pretreatments compared to the as-received condition, as shown in Figure 9. Sandblasting increased the shear strength by 20%, anodizing by 16% and laser structuring by 14%. The standard deviation for the as-received shear tensile tests is the highest compared to the textured metal surface shear tensile tests. Sandblasting produced the lowest standard deviation of results, followed by anodizing and laser structuring.

3.4. Wrinkle height
In terms of wrinkle height, the surface textures had different effects, which can be seen in Figure 10. An anodized surface, for instance, had a negative effect on the flow behavior of the epoxy resin. While wrinkles with a height of $h_{wr} = 0.52$ mm can be observed at a measuring depth of $z_{md} = 2$ mm, they decrease steadily to $h_{wr} = 0.16$ mm at $z_{md} = 17$ mm, increase again and then decrease to a value of $h_{wr} = 0.12$ mm at $z_{md} = 27$ mm. In contrast, treatment of the sheet blanks by sandblasting and laser structuring resulted in lower wrinkle formation. Laser structuring yielded wrinkles with a height of approx. $h_{wr} = 0.40$ mm at a measuring depth of $z_{md} = 2$ mm, which decrease to a value of $h_{wr} = 0.09$ mm at $z_{md} = 17$ mm. The further course is almost analogous to the reference values. Sandblasting had the greatest positive influence of the three methods used. On these test specimens, wrinkles with a height of approx. $h_{wr} = 0.41$ mm were detected $z_{md} = 2$ mm, which, however, decrease with the largest
drop to a value of \( h_{wr} = 0.04 \text{ mm} \) at \( z_{md} = 17 \text{ mm} \). Although this drop also occurs in the sandblasted specimens, it is much shallower, as at a measuring depth of \( z_{md} = 22 \text{ mm} \), the wrinkles had a height of \( h_{wr} = 0.08 \text{ mm} \).

**Figure 10:** Height of the wrinkles \( h_{wr} \) above the measurement depth \( z_{md} \)

### 4. Discussion

After anodizing, the surface roughness decreased as the topography of the starting material was slightly smoothed. The nanoporosity induced by the formation of porous zinc oxide could not be measured by measurements with the tactile roughness tester. However, the adhesion properties in the shear tensile test were improved compared to the as-received condition. This is probably due to the additional mechanical interlock between the steel substrate and the adhesive, as the adhesive flows into the pores during joining and cures there, which was already observed in an earlier study by Engelkemeier et al. [10]. Furthermore, sandblasting and laser structuring also led to a significant improvement of the adhesive properties. While the highly deformed sandblasted surface is ideal for creating a mechanical bond between the adhesive and the substrate, laser structuring results in surface enlargement at both the macroscopic and nanoscopic levels. A combination of the two methods has a positive effect on the adhesive properties as well. Although the results of the shear tensile tests with double or single overlapped shear tensile specimens should be taken with caution, since they do not represent a realistic load case of FML components, they are suitable for a qualitative statement on whether the surface pretreatment adds value compared to the untreated surface. [17]

Despite the improved adhesion properties of the anodized zinc coated steel sheets, wrinkling during deep drawing of the FML parts could not be reduced. In the range from \( z_{md} = 2 \text{ mm} \) to \( z_{md} = 7 \text{ mm} \) it even increased in comparison with the as-received condition.

However, it may be worthwhile to look for correlations between the surface roughness values and the wrinkle height, as sandblasting yielded both the roughest surface and the best wrinkle height, and laser structuring slightly increased the roughness value while decreasing the wrinkle height. It appears that macroscopic surface enlargement, as occurs with sandblasting and to some degree with laser patterning, is beneficial in preventing wrinkling due to accumulation of the matrix material. In contrast, nanoscopic surface enlargement, as in anodizing and to some extent in laser structuring, is ineffective for improving the accuracy of deep-drawn FML parts. Furthermore, it should be considered that especially with laser ablation, the parameterization is extremely complex and at the same time has a great influence on the resulting surface properties. Thus, it has not been conclusively determined that
laser structuring could not achieve similar results to sandblasting. For this purpose, further parameter studies should be carried out with regard to laser structuring.

5. Conclusions
In this study, the influence of the surface pretreatments sandblasting, anodizing and laser structuring on the adhesion properties between components in FML parts was investigated. Subsequently, the components were formed to test whether the surface pretreatment also has a positive effect on the manufacturing accuracy.

- Surface roughness is greatly increased by sandblasting and slightly increased by laser structuring. It is slightly reduced by anodizing.
- The three processes significantly improve the adhesion properties. With the parameters used in this study, the best results were achieved by sandblasting.
- The development of the wrinkle height is analogous to the surface roughness. The lowest wrinkle heights were measured after sandblasting, while laser structuring only slightly reduced the wrinkle height and anodizing slightly increased it.
- A direct correlation between surface roughness, i.e., macroscopic surface morphology, and wrinkle obstruction is possible and should be further investigated.

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