Minimum invasive production-related SLS specimen manufacturing for interface characterization of hybrid materials made by RTM

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Abstract

This work shows a new way of single lap shear specimen production for hybrid metal-composite materials, which can be used to characterize process-induced interface property variations. Due to this special procedure, the specimen manufacturing can be done production-related and with a homogenization of the thermal stresses occurring in the joint area of the hybrid at each specimen. The exemption process of the specimen is kept in a minimum invasive way, not affecting the interface in the tested zone. The influence of the metal surface structure specifically created by micro form milling as well as the influence of the curing temperature on the maximum shear strength of the interface, were investigated. Finally, the driving failure mechanisms were identified and described.

Keywords Hybrid metal-CFRP composites · Interface characterization · Single lap shear test · Resin transfer molding · Processing of composites

1 Introduction

Today, hybrid materials and structures are an upcoming and promising development in research as well as in automotive and aeronautic industry. Such hybrids combine different material types in one part and try to take advantage of their different properties [1]. Therefore, the interface zone between the two materials has a significant influence on the performance of the final hybrid part [2]. Depending on the processing route, the interface is either built in an independent step or while one of the materials is built [3]. The latter case can be applied to fiber-reinforced plastics (FRPs) which can be hybridized with metals for instance. The injection phase of the resin transfer

molding (RTM) process can be used to combine the manufacturing step of the FRP and the joining step of the metal and FRP component [4–6]. Therefore, the fiber preform and the metal component are placed together in the mold. The resin system is injected into the mold and acts as the matrix for the FRP as well as an adhesive towards the metal component. It is important to minimize the formation of voids during this step as they directly influence the quality of the final part as well as the interface strength of the hybrid part [7–9]. In addition, the surface of the metal structure has a big impact on the interface strength [8, 10]. Therefore, it is important to functionalize the surface to improve the performance of the manufactured components. For the fabrication of micro textures and micro features on metallic surfaces, several technologies, such as laser processing, etching, electrochemical- and electro discharge-micromachining, are used [2, 11]. In addition, mechanical processes, like milling processes, can obtain a high form accuracy and a good surface quality of manufactured micro features with efficient productivity [12]. Xie et al. used a micro grinding process with a diamond wheel V-tip to generate micro cutting-edge arrays on ball cutters as shown on the right side of Fig. 1. By applying these functionalized tools to a 5-axial milling process, microscale pyramid patterns as displayed at the left side of Fig. 1 could be generated on aluminum and die steel freeform surfaces [13].

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In order to quantify the interface strength of polymers and FRPs, a wide range of test methods and standards in the field of shear testing are available. Besides the procedure for the determination of the adhesive strength, further aspects such as surface preparation and adhesive environmental durability are considered [14–19]. Various lap joint configurations are available and can be ranked by increasing joint improvement as follows: single lap, double lap, single cover plate, and double cover plate [20]. Due to rather uncomplicated sample preparation and testing procedure in a simple monotonic tensile test, the lap shear test is a preferred and widely used method for shear strength testing of adhesively bonded joints [3, 21, 22].

In the case of the single lap shear (SLS) configuration, the test specimen consists of two rectangular sections with an overlapping area, large enough to provide failure in the adhesive and not in the substrate. The lap shear strength (adhesive strength) determined in subsequent tensile testing is given by:

$$\tau = \frac{P}{bL}$$

where P is the maximum load, b is the joint width, and L is the joint length.

Nevertheless, there are a number of influencing parameters for the determination of shear strength values, primarily referring to the specific procedure of sample preparation, concerning surface treatment as well as adhesive application and thickness [23]. The analysis regarding “pure shear” in a single lap shear setup assumes that the joint is rigid, and that the adhesive only deforms in shear. In fact, the resulting stress distribution, across and along the bond length is complex. The sample-dependent eccentricity of the load path causes out-of-plane bending moments, resulting in bending deformation in the overlapping regime, along with additional peel stresses in the adhesive layer. To lower the influence of these effects, which effectively reduce the structural efficiency of the joint, various types of specimen configurations are available in order to change the failure mode from peel towards shear [24–27].

2 Experimental procedures

For manufacturing the hybrid metal-CFRP SLS specimens, a four-step procedure was carried out. At first, the surface of

each metal component was sand-blasted to ensure a higher level of surface roughness. Three sets, each consisting of 16 metal parts, were processed: the first set was kept to produce reference specimens, the second set was micro-structured by a milling cutter with a cutting-edge point angle of 28° , and the last set was micro-structured by a milling cutter with a cutting-edge point angle of 45° (see following section). This enlarges the surface and enables mechanical interlocking towards the CFRP component. In a third step, the RTM technique was used to produce the hybrid metal-CFRP specimens. There, the metal parts were inserted into a fluorocarbon elastomer (FPM) template, the fiber reinforcement preform was placed on top, and then the resin system was injected. Finally, the specimens were cut out from the produced plate and a groove was incised into the samples to create an SLS specimen. For this step, only the CFRP component needs to be cut, as the FPM can be removed beforehand. This reduces the heat generated during the cutting, which preserves the interface from damage. The novelty of this method is presented in Fig. 2, where the standard procedure and the procedure shown in this work are compared. In the standard procedure, the interface between the two components is built in a separate gluing step, while in the novel approach, it is realized during the RTM process. This makes the procedure much more production-related for the one shot hybrid (OSH)-RTM process [4–6].

2.1 Metal component preparation

In order to generate a pattern of defined grooves in the shear zone area of the SLS specimens, uncoated carbide inserts have been modified by the use of a wire-cut electrical discharge machining (EDM)-process performed with a wire of 0.25 mm diameter. As part of this modification, single micro cutting-edges were prepared at the tip of the cutting inserts as shown in Fig. 3.

Two different inserts with cutting-edge point angles of 28° and 45° were manufactured and used for milling operations to create the desired surface textures. The milling cutter equipped with the form shaped insert (28° or 45° point angle) was attached to a C20U 5-axis machining center of the company Hermle AG (Gossheim, Germany) and single grooves were generated. Therefore, a line pitch of 0.3 mm, a cutting depth of $a_p = 0.03$ mm, a feed rate of $v_f = 10$ mm/min, and a

Fig. 1 Microscale pyramid patterns (left) and ball cutters with micro cutting-edge arrays (right) [13]

Fig. 2 Comparison of the processing steps for manufacturing and testing hybrid metal-CFRP SLS specimens following the standard procedure and the novel approach presented in this work

spindle speed of $n = 730$ rpm were used as machining parameters. Figure 3a shows the surface texture of a specimen machined by the tool with a cutting-edge point angle of 28° . As depicted in Fig. 4b, single grooves with a nearly consistent depth of around $50 \mu\text{m}$ represent a homogeneous texturing result.

2.2 Hybrid CFRP steel plate manufacturing

As already shown in previous studies the difference in the thermal expansion coefficients of CFRP and steel leads to deformations of hybrid plates [5, 28]. To reduce the resulting stresses and deformations, an uncoupling approach was used for this study. A 2-mm thick sheet of FPM was used as a template for the metal inserts. This reduces the area of the hybrid and decouples the occurring stresses in the bonded areas. Figure 5 shows the lower mold part with the entire material stack used for the manufacturing of the hybrid specimens and the FPM template with the metal inserts beside it. Table 1 gives an overview of the materials used for this work.

Two plates with the dimensions of $270 \times 270 \times 4$ mm were produced at two different levels of curing temperature. Table 2 lists the processing parameters for manufacturing of each plate.

The steel mold was heated by a water-based heating system of the type Thermo-5 HB-180Z2 from HB-Therm (St. Gallen,

Switzerland). An ACMOScoat 82-1181-71 release agent from ACMOS Chemie KG (Bremen, Germany) was applied to the mold prior to the manufacturing step. A LZT-OK-80-SO press of the company Langzauner (Lambrecht, Austria) was used to mount the mold and to apply a compressive load of 300 kN throughout the experiment. For the resin injection, a Nodopur VS-2K injection unit from Tartler (Michelstadt, Germany) was used. After 25 min of curing the plate, containing the 24 hybrid metal-CFRP specimens, was demolded and cooled down to ambient temperature (23°C) by free convection.

2.3 Specimen cutting

Subsequent to processing the hybrid metal-CFRP plate, the SLS specimens were prepared. In a first step, one center hole of each specimen, already pre-machined in the steel part of the hybrid and shown in Fig. 6a, was drilled through the CFRP layer. The drilling was done with the steel side up as the drilling direction might affect the interface otherwise [29]. In reference to this hole, the second center hole of each specimen was machined on its defined position using a vertical NC-machining center VF-3 of the company Haas (Oxnard, USA). After the individual specimens were cut out of the plate by the use of a band saw, the drilled holes were used to fix the specimens by centering screws, CFRP side up, at a defined position to a work piece holder. Finally, finish machining,

Fig. 3 Functionalized tool. **a** Milling cutter with structured cutting insert. **b** Cutting insert tip with single micro cutting-edge with a point angle of 28°

Fig. 4 Texturing result. **a** SLS specimen and 3D-measurement of the textured surface in pseudocolor visualization. **b** Profile extracted across the created micro grooves

consisting of a trimming process to generate the outer contour of the specimen and a cutting process to generate the groove in the CFRP layer, took place (see Fig. 6b). The trimming and cutting processes were done with diamond-coated cemented carbide tools. The cutting parameters of these finishing processes are given in Table 3. To exclude influences of cooling lubricant [30] onto the hybrid structure, the processes were performed under dry conditions.

2.4 SLS testing

In order to determine the interface properties of the metal-CFRP hybrid composite, SLS tests were performed. The SLS specimens with dimensions of $80 \times 15 \times 4$ mm and an

overlapping area of 10×15 mm were mounted between the standard grips of the tensile testing device ZWICK Z010 (Ulm, Germany). The tests were performed in standard climate (23°C , 50% r. H.) with a test speed of 1 mm/min until fracture. Shear strength values were determined based on the maximum force values detected by a load cell with a capacity of 10 kN, related to the bonded area.

3 Results and discussion

In order to investigate the influence of the surface treatment and the curing temperature on the shear strength of the hybrid bonding, an analysis of variance (ANOVA)

Fig. 5 Lower mold with the material stack used for specimen manufacturing. **a** GF, **b** CF Style, **c** CF 650SA4, **d** Steel, **e** FPM

Table 1 Materials used for specimen manufacturing

Material	Specification	Supplier	Areal weight [g/m ²]	Symbol
Glass fibers (GF)	Microlith ST 3022	Johns Manville Corp.	27	a
Carbon fibers (CF)	Style 423–1, 6 K	ECC	253	b
Carbon fibers (CF)	650SA4, 12 K HS	Chomarat	670	c
Metal	Steel 1.0548 HC340LA	TK	–	d
FPM	Viton	DuPont	–	e
Resin	Epinal IR 77.55-A1	bto-epoxy	–	–
Curing agent	Epinal IH 77.55-B1	bto-epoxy	–	–

Table 2 Processing parameters for the two different plates

Set Nr.	Resin temp.	Mold temp.	Mass flow	Max. injection pressure
	[°C]	[°C]	[g/min]	[bar]
#1	65	100	150	20
#2	65	120	150	20

was carried out. Therefore, six cases of specimens were studied: two plates with the different processing parameter sets shown in Table 2 were produced containing metal inserts with the three different surface preparations (sand blasted (SB) and micro milled with 28° and 45° cutting-edge point angle). To randomize the effect of the position of the specimen on the plate, the metal inserts were placed in the FPM template as shown in Fig. 7.

The shear strength results of the different SLS specimens are displayed in Fig. 8. The micro grooved specimens performed much better than the specimens with the sandblasted steel surface. Furthermore, a higher curing temperature shows increased shear strength. While the sandblasted reference specimens produced at 100 °C curing temperature show a

mean shear strength of 14.98 MPa, this value is improved by over 15% to 17.67 MPa for the specimens produced at a curing temperature of 120 °C. Similar improvements are observed for both micro grooved specimen types. These specimens perform with shear strengths of 17.99 MPa (28°) and 17.50 MPa (45°) after 25 min curing at 100 °C. The shear strength values increased again by over 15% by lifting the curing temperature to 120 °C, which results in maximum shear strengths of 21.21 MPa (28°) and 21.00 MPa (45°) respectively.

This means an improvement of the interface strength can be achieved by applying micro grooves on the metal surface as well as by raising the curing temperature. Both effects show similar improvements on the shear strength of the hybrid material. This can clearly be seen at the main effect diagram shown in Fig. 9. While the standard deviation of the shear strength at the two different levels of curing temperature is quite similar, this is not the case for the different types of surface preparation.

The lowest standard deviation of the shear strength occurred at the sand blasted surface, followed by the specimens with the surface pre-treated with the 45° cutting insert and the highest standard deviation of the specimens prepared with the 28° cutting insert. One explanation

Fig. 6 Sample manufacturing. a SLS specimens before the final machining step. b Machining strategies for the trimming and groove cutting operation in CFRP

Table 3 Machining parameters for sample manufacturing

Operation	Tool diameter	Feed rate	Feed per tooth	Cutting speed
	[mm]	[mm/min]	[mm]	[m/min]
Trimming	10	509	0.05	80
Groove cutting	2	288	0.01	60

could be the influence of stress concentrations occurring in the interface zone at the grooves and peaks of the metal. The sand-blasted metal surface shows a uniform level of roughness, which leads to a uniform stress distribution over the whole surface. The edges of the metal surface cut with the 45° insert show less sharp edges compared to the surface cut with the 28° insert. This leads to higher local and non-uniformly distributed stress concentrations, which in turn would lead to earlier failure.

However, this effect is accompanied by the effect that the grooves with 45° opening angle cannot transfer the same force into the material. This is shown in Fig. 10 as the force vector for the 28° groove is 88% longer than the force vector for the 45° groove. In addition, these two effects result in a slightly higher shear strength of the hybrid metal-CFRP SLS specimens produced with a metal surface cut with 28° accompanied with a higher standard deviation.

In order to study the influence of the curing temperature on the interface strength, a series of tensile tests on neat resin specimens were performed. The curing states of the resin were provided at 100 °C and 120 °C, for a curing time of 25 min. According to the ISO 527-2 standard, dumbbell tensile test specimens were cut out of the cured plates, five specimens for each curing temperature.

Fig. 7 Positions of the specimens on the produced plate indicating the surface treatment with sand blasting (SB squares), 28° (triangles), and 45° cutting-edge point angle (circles)

The tensile properties were determined by the use of a tensile testing device ZWICK Z010 (Ulm, Germany) with a load range of 10 kN combined with optical extensometer ARAMIS – 3D-Digital Image Correlation from GOM GmbH (Braunschweig, Germany) for the displacement measurement. In Fig. 11, the resulting tensile properties of the two different resin states cured at 100 and 120 °C are compared regarding Young's modulus at a testing temperature of 23 °C at 50% r. H.

As one can see, there is an unexpected decrease of the tensile modulus at the increased curing temperature level. The resin cured with 100 °C reaches a modulus of 3100 MPa while the higher curing temperature of 120 °C decreases the modulus to 2870 MPa. This effect is related to the resin formulation which was specifically developed for adhesively bonding metal and CFRP parts during the RTM process. However, literature shows that adhesives with higher modulus lead to higher shear stress concentrations on the edges of the bonded areas [31–33]. This explains the higher reachable shear

Fig. 8 Maximum shear strength of the SLS test by different settings

Fig. 9 Main effect diagram showing the influence of the surface treatment and the curing temperature on the shear strength of the interface between steel and CFRP

strength of the hybrid which was cured with higher temperature.

Nevertheless, there is also the effect of thermal stresses, which are induced by the different thermal expansion coefficients of steel and CFRP. This would reduce the maximum reachable shear strength in the bonded area. Due to the special manufacturing of the SLS specimens, which are decoupled by the elastomeric template, the areas of the two bonded materials are kept small, which in turn leads to a lower level of thermally induced stresses. Figure 12 outlines schematically the resulting shear stresses, which occur while testing the specimens, dependent on the curing temperatures and the resulting Young's modulus of the resin. Due to the statement above, these shear stresses can be superposed, which results in the addition of thermally induced stresses at a low temperature

gradient and the high shear stresses occurring due to the higher modulus of the resin and vice versa.

All of the effects described here can also be seen in the fracture pictures shown in Fig. 13. The sand-blasted surfaces show only small areas of cohesively bonded resin on the metal surface, which leads to lower shear strength. Furthermore, the surface prepared with 28° cutting-edge point angle shows more broken resin parts than the one prepared with 45° cutting-edge point angle. This is because of higher normal forces acting on the resin peaks, which are shearing them off at the ground.

The fracture of the interface always started next to the gap between the metal sheets. This can be explained by the S_{33} peel stresses occurring at this point [34]. The S_{33} stresses are shifted towards this side as the Young's

Fig. 10 Force transferrable by 28° and 45° cut grooves

Fig. 11 Comparison of tensile modulus of neat resin after 25 min curing with 100 °C and 120 °C

modulus of the CFRP is lower than the one of the used steel sheet.

4 Summary and conclusion

This work shows a way of producing hybrid SLS specimens containing CFRP and steel by using the RTM technique. The shown procedure can be applied to any other hybrid material combination containing FRP and a second rigid material as long as the RTM technique is used. An advantage of this procedure is that the specimens are manufactured in a production-related manner and in a minimum invasive way. Therefore, the results of the shear strength measurements are much more

representative for real components. Due to the used elastomeric template, the thermal stresses occurring in the bonded areas can be decoupled. To prove, that this procedure is able to reveal differences in the interface strength occurring due to different surface structures and curing temperatures, two plates were manufactured and 48 SLS specimens were tested. To ensure that the interface is not damaged during the exemption process, special tools and sets of machining parameters were used. Furthermore, the used FPM template prevents the interface from damage because only the CFRP component needs to be cut. It was found that a surface structuring by micro form milling is advantageous compared to simple sandblasting of the surface. Dependent on the cutting-edge point angle, slightly different levels of maximum shear strength were found. The failure of the SLS specimen is mainly induced by stress concentrations, which result from the peaks produced by the milling process or by a Young's modulus of the resin system which is too high. Therefore, it was shown that the highest shear strength could be reached by using a high curing temperature and a cutting-edge point angle of 28°. This result is also driven by the aspect that the resin system has a reduced Young's modulus at the elevated curing temperature, which reduces the stress concentrations in the edge areas of the hybrid joint.

Therefore, it can be concluded that:

- The shown procedure is able to produce a high amount of production-related manufactured SLS specimen in relatively short time,
- The manufactured specimen can reveal process induced interface variations,
- Structuring of the metal surface component of the hybrid composite is positively affecting the interface strength and therefore improving the properties of the whole hybrid part, and
- The increase of the maximum interface strength achieved by the increase of the curing temperature is independent from the metal surface preparation.

Fig. 12 Resulting shear stresses under tension of different manufacturing sets

Fig. 13 Fractured surfaces of the SLS specimens referred to the structured surfaces and curing temperatures of the related CFRP and steel side

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflicts of interest.

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