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CHARACTERIZATION OF INTERFACE PROPERTIES OF FIBER-METAL-LAMINATES (FML) WITH OPTIMIZED SURFACES

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ABSTRACT: Fiber-Metal-Laminates (FML) combine the outstanding specific mechanical properties of carbon fiber reinforced polymers (CFRP) and lightweight metals. Additionally they promise exceptional dynamic properties. However, laminates are prone to fail prematurely due to delamination. The resistance to delamination of a FML can be characterized by determining the interlaminar shear strength and shear energy. The mechanical properties can be increased by optimizing the laminate surfaces to increase adhesion between the composite partners.

The surfaces were optimized by plasma enhanced chemical vapor deposition, by sand blasting and by grinding. Additionally an elastomer interlayer was introduced to increase adhesion and to endure high strains. These surface modifications were compared to a reference FML without modified interfaces. The characterization of the laminates was carried out using the edge shear test to enforce failure due to shear load.

This contribution focuses on the characterization and optimization of the adhesion between composite partners in a laminate. The authors present results of surface optimization in Fiber-Metal-Laminates.

KEYWORDS: Fiber-Metal-Laminate, Interface Characterization, Interlaminar Shear Strength, Materials Testing

1 INTRODUCTION

Lightweight solutions are often based on an extraordinary stiffness to density ratio, but dynamic resistances [1], such as low crack propagation [2] are, depending on the application, even more important. Fiber-Metal-Laminates combine high crack resistance with low density and are already commercially available (c.f. GLARE [3,4]). High specific quasi static properties were prerequisites for the laminates investigated in this study, even though the focus was on interlaminar properties.

The performance of the commercially available composite structures can be optimized by replacing the glass fibers with carbon fibers due to the higher stiffness and strength, yet lower density of the carbon fiber. However the replacement introduces risk of contact corrosion as well as sensitivity to delamination caused by very different coefficients of thermal expansion (CTE-mismatch) [5].

The addition of a surface layer with passivating properties and optimized adhesion can improve the laminate. It may prevent corrosion by electrical decoupling and counteract delamination by increased adhesion. In this study the metal was coated by plasma enhanced chemical vapor deposition or mechanical treatment was carried out. Additionally the application of an elastomer layer was implemented in the production process. The adhesion of the laminate layers was tested by the edge shear test [6] to characterize the adhesive bond between the carbon fiber reinforced polymer and the metal. The adhesion tested accounts for the resistance to delamination, which is of great interest when testing laminates, as laminates often prematurely fail due to delamination. To inhibit delamination adhesion is to be increased by optimization of the CFRP/metal interface. The maximum shear stress [7] to separate the laminate layers and the energy dissipated will be characteristic values for the adhesion of the laminate.

2 MATERIALS

The Fiber-Metal-Laminates consist of a Carbon Fiber Reinforced Polymer (CFRP) and either an aluminum alloy or a steel layer.

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The FML was manufactured at the Institute for Production Science (wbk) at KIT. A Lauffer type RP 400 OK 920 machine press was used at 150 °C at a pressure of 23 bar for the curing cycle of 5 minutes.

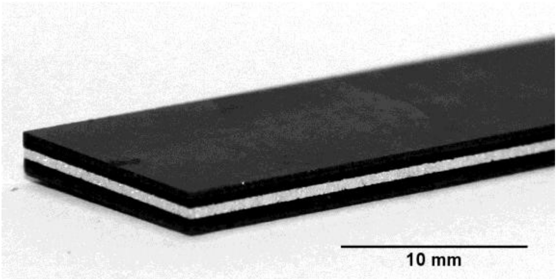

Fig. 1 *Fiber-Metal-Laminate with elastomer interlayers*

Fig. 1 shows a FML with elastomer interlayers where the layer succession is CFRP – Elastomer – Aluminum – Elastomer – CFRP. This sequence is used for dimensional stability as the CTE-mismatch does not deform the FML. The aluminum can be substituted for a steel layer and the elastomer can be replaced by a CVD coating.

2.1 CARBON FIBER REINFORCED POLYMER

The CFRP used in this study is a Hexcel M77/42%/UD90/CHS with unidirectional high strength carbon fibers and an optimized resin for fast curing cycles to counteract the usually long curing cycles of FML. For the application in the FML a low sheet thickness of 0.1 mm was essential to guarantee a variable layup of the CFRP layer in the Fiber-Metal-Laminate.
The curing temperature of the epoxy matrix was matched to the curing temperature of the elastomer interlayer to ensure the best quality FML with elastomer interlayers.
The CFRP has high specific mechanical properties, because of the high strength fibers and a fiber volume content of 50 %, resulting in a density of 1.5 g/cm³. The unidirectional prepreg sheets were arranged to a biaxial CFRP layer. The recommended curing cycle for this prepreg is 2 minutes at a temperature of 150 °C and a pressure >5 bar.

2.2 METAL

The metal used in the FML was chosen according to high resistance to sheet bending and simultaneously low density. Another restriction was the possibility to roll the metal to a 0.3 mm thin sheet. Therefore the first choice was an aluminum alloy with high strengths.
For high overall mechanical properties disregarding the density restriction of the metal another FML with a steel sheet was fabricated. Steel was used to vary chemical and mechanical properties on top of the good rollability and availability.

2.2.1 ALUMINUM 2024-T3

Structural applications often use the 2024 alloy in a hardened state. An aluminum 2024-T3 was chosen to ensure comparability as many laminates use this alloy [3,4,7]. The alloy has extraordinary specific strengths and good availability in rolled thin sheets. The minimum thickness of 2024-T3 commercially available is 0.3 mm, being sufficient for the FML.

2.2.2 STEEL TS275

The steel chosen was a carbon steel, as other steels with higher strengths are not commercially available in the desired thickness. The results obtained with this steel can be transferred to other alloys as the surface properties do not differ greatly when the alloy is changed.

3 SURFACE OPITMIZATION

To counteract premature failure due to delamination between laminate layers the adhesion has to be enhanced by surface optimization.
The optimization may solve the problems caused by the mismatch of thermal expansion coefficients (CTE-mismatch) and corrosion potentials caused by the combination of CFRP and metal sheets.
In this research plasma enhanced chemical vapor deposition, an elastomer interlayer and mechanical treatment will be compared to the reference FML without any interface customization.

3.1 PLASMA ENHANCED CVD

Plasma enhanced chemical vapor (PECVD) deposition was used to deposit a silicone-based coatings on the metal layer. Different kinds of layer compositions were used and are described below.
PECVD was performed by Fraunhofer Institute for Chemical Technology (ICT) in Pfinztal, Germany.
Bulk SiO_2 was used as a surface optimization layer and as a base layer for different PECVD coatings to ensure better adhesion to the metal substrate. The silicone-based layers are desired to inhibit corrosion.
The contact angle of the coating was measured at Fraunhofer ICT and is expected to define the adhesion of the laminate layers. The surface property can be chosen from hydrophobic to super hydrophilic. The hydrophilic layers have a contact angle of 50° on steel and 67° on aluminum. The hydrophobic layer has an angle greater than 90° and the super hydrophilic layer's contact angle is 0°.

Table 1: *Properties of PECVD surface optimization*

Surface	Properties	Thickness
Bulk SiO_2	hydrophilic	1.2 µm
$SiO_2/Si_wC_xH_yO_z$	hydrophobic	1.1 µm

nanoporous	Super hydrophilic	100-200 nm
SiO_2/nanoporous	Super hydrophilic	1.4 µm

3.2 MECHANICAL TREATMENT

Mechanical treatments for enhanced adhesion of metals to CFRP are based on the principal of form fit. One treatment was grinding the metal layer and the other was sand blasting. Both treatments are predicted to increase adhesion due to the roughened surface.

3.3 ELASTOMER INTERLAYER

The elastomer used in this research was provided by Kraiburg, the registered trade name is Kraibon and it is optimized for applications with CFRP. The mixture chosen, SAA-9579/52, does not only possess enhanced adhesion to CFRP with epoxy matrix but also to aluminum.

The elastomer interlayer promises inhibited corrosion, balancing of CTE-mismatch and increased adhesion. The elastomer layer has a thickness of 0.5 mm and a curing temperature fitted to established CFRP curing temperatures. The curing cycle for the FML is defined by the elastomer, as it is the slower component of the fiber-metal-laminate. The elastomer requires a curing cycle of 5 minutes at >4.8 bar pressure at the given 150°C. Therefore the cycle time is set to 5 minutes at the temperature defined by the CFRP of 150°C.

4 CHARACTERIZATION

The fiber-metal-laminates were characterized using the edge shear test [6] to induce pure shear into the interface. This test was deliberately chosen for these experiments, because the interface can be tested without any interference of the laminate components.

Additionally the specimen had a simple geometry and was small compared to the double notch test [7], allowing a greater number of tests to statistically ensure the results. The 20 x 10 mm² specimens were cut by water jet cutting. The exact dimensions were measured using a micrometer screw. Stresses were calculated using the measured cross section.

The test rig prevents buckling by supporting the specimen during the experiment (c.f. Fig. 2). Also the shear plane can be deliberately put into the interface of the laminate structure to force failure in the desired plane.

Fig. 2 FML with elastomer interlayer in the edge shear test

The experimental set-up induces pure shear stress into the specimen using two edges (c.f. Fig. 2), hence the name edge shear test. The set-up was mounted on a ZwickRoell ZMART.PRO 100kN universal testing machine. The test was carried out with a speed of 1 mm/min to achieve quasi static testing conditions. The tests were performed at room temperature and the interface failed under shear stress. The shear stress is the quotient of the maximum force measured in the experiment and the area of the sample measured before testing. The shear energy is the integral under the stress-strain curve of the edge shear test. All specimens showed comparable stress-strain curves, except the FML with elastomer interlayers. Two representative curves are shown in Fig. 3.

Since the elastomer interlayer alters the interface characteristics drastically the shear strength alone is not significant enough to solely characterize the FML and rank the surface modifications. Therefore the shear energy is considered as well. Fig. 3 shows the two types of stress-strain curves, one being a stiff and brittle and the other being soft and ductile.

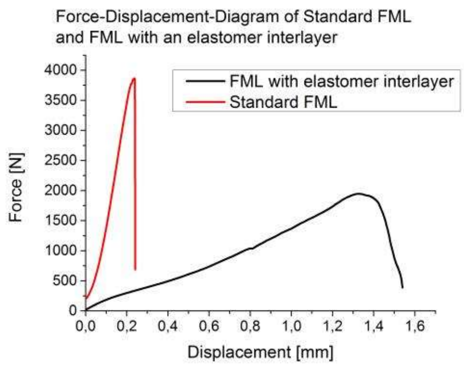

Fig. 3 *Force-displacement curves of Standard FML compared to FML with elastomer interlayers*

4.1 SHEAR STRENGTH

The shear strength of the laminate shows the maximum shear stress value and characterizes the adhesion strength when loaded with shear stress. FML with modified surfaces are desired to have higher strengths compared to the reference FML.
FML with shear soft interfaces such as the elastomer interlayer are expected to have a lower shear strength compared to stiffer layers like the PECVD coatings.
The shear strength for lightweight applications should be divided by the laminate density. To ensure good comparability between all laminates, the absolute values are presented here.

4.2 SHEAR ENERGY

The shear energy is equal to the area under the stress-strain-curve. It is the energy needed to separate the layers of the laminate, which equals the energy dissipated during delamination.
For stiff layers with high strengths and low strains the total energy is expected to be lower compared to the elastomer layers which have lower strengths but much greater strains (c.f. Fig. 2).
The shear energy is a critical value when describing interface properties.

5 RESULTS

All specimens showed failure due to shear stress in the interface between the metal sheet and the CFRP. A total of 16 specimens were tested for every modification of the laminate to generate one value for shear strength and shear energy.
The results of the optimized surfaces tested by the edge shear test will be compared to the reference FML without modified interfaces.

5.1 REFERENCE FML

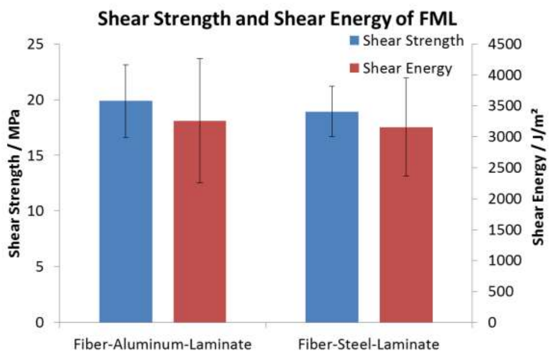

Fig. 4 *Interface properties of FML using Aluminum and Steel substrates characterized by the edge shear test*

The mechanical interface properties for the reference laminates are shown above (Fig. 4).
The shear strength and energy generally depend on the metal used in the laminate. The metals used for this research, Al 2024-T3 and Steel TS275and their surface properties with unmodified surfaces do not differ greatly (c.f. Fig.4). The interface properties are marginally lower using steel sheets. The exact values for the interface properties can be found in Table 1 for aluminum and Table 2 for steel.
The surface optimization, however, greatly depends on the metal layer and therefore the results are compared to the laminate with the same metal substrate and unmodified surface. This specimen is called “reference” sample for the respective metal. The densities of the laminates are not accounted for in these results, but it the higher density of steel has to be kept in mind when comparing FML with aluminum and steel layers.

5.2 QUALITATIVE ANALYSIS

The introduction of an adhesive coating does not change the mechanical behavior of the laminate, because all samples had the characteristics of the red curve in Fig. 3.
This statement does not include the elastomer interlayer, which allows large strains and lowers the shear strength. The characteristic curve for this configuration can be found in the black graph in Fig. 3.
This behavior cannot easily be compared to the reference sample. The interface properties of the reference and the elastomer layup are shown in Fig. 5. The aluminum-based FML is shown here, but the steel substrate indicates the same behavior.

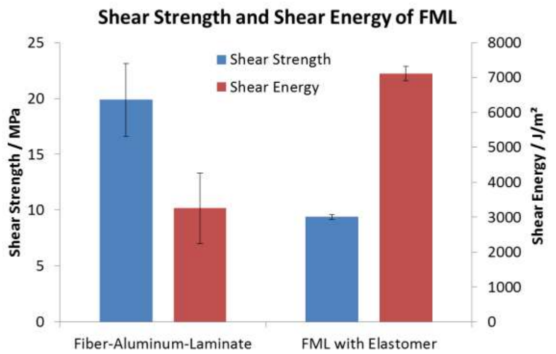

Fig. 5 *Interface properties of FML with an elastomer interlayer compared to the reference*

As predicted the shear strength of the FML with elastomer is reduced to approximately half of the reference’s strength. The elastomer also causes the shear energy to more than double in value compared to the FML.
Additionally the size of the error indicators shrinks when the elastomer interlayer is applied.

5.3 MECHANICAL CHARACTERIZATION

For each surface optimization 16 samples were tested and the shear strength and shear energy was calculated. The order is according to the succession in Chapter 3. The results are arranged by metal substrate, first the aluminum, followed by the steel substrate.

Table 2: *Mechanical characterization of the surface optimization of Fiber-Aluminum-Laminates*

Surface	Shear Strength MPa	Shear Energy J/m²
Reference	19.9 (± 3.3)	3260 (± 1006)
Bulk SiO_2	20.9 (± 2.7)	3037 (± 559)
$SiO_2/Si_wC_xH_yO_z$	16.2 (± 3.4)	2284 (± 1266)
nanoporous	10.9 (± 3.3)	1590 (± 521)
SiO_2/nanoporous	16.5 (± 2.7)	2447 (± 585)
Grinding	17.6 (± 2.7)	2994 (± 691)
Sand blasting	22.6 (± 2.4)	2757 (± 500)
Elastomer	9.4 (± 0.2)	7108 (± 206)

The results shown in Table 2 show the values of shear strength and shear energy of all surface modified FML. Each value is supported by 16 test specimen and is therefore statistically verified.
An increase in shear strength and shear energy would be desirable, but could not be reached by any modification.
A decrease in shear strength can be seen by most surface modifications compared to the reference FML. The only substantial shear strength increase was reached by sand blasting, but is paired with a reduction in shear energy.
The elastomer reduces shear strength and increases shear energy drastically.

Table 3: *Mechanical characterization of the surface optimization of Fiber-Steel-Laminates*

Surface	Shear Strength MPa	Shear Energy J/m²
Reference	18.9 (± 2.3)	3158 (± 788)
Bulk SiO_2	12.0 (± 2.1)	1068 (± 534)
$SiO_2/Si_wC_xH_yO_z$	9.1 (± 1.7)	555 (± 191)
nanoporous	4.1 (± 0.9)	460 (± 53)
SiO_2/nanoporous	14.1 (± 2.0)	1287 (± 318)
Grinding	15.4 (± 3.3)	2870 (± 1001)
Sand blasting	29.4 (± 1.2)	9026 (± 1254)
Elastomer	9.4 (± 0.5)	6826 (± 391)

The values for strength and energy in Table 3 show that only sand blasting increased the shear strength and sand blasting and the elastomer interlayer raised the value for shear energy.
The PECVD coatings lowered both values of the interface properties compared to the reference.

5.4 CONTACT ANGLE MEASURMENT

A contact angle measurement was carried out post mortem to determine, if the adhesion of the coating to the metal failed.

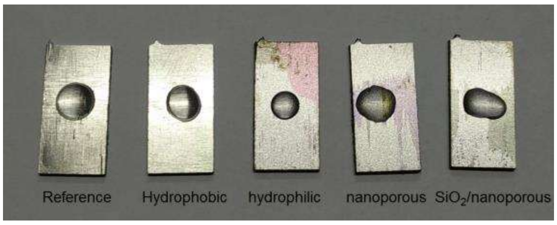

Fig. 6 *Contact angle experiments on failed FML specimens*

Fig. 6 shows, that the contact angle of all metal layers are similar to the reference. The contact angle of the coating cannot be seen anymore.

6 DISCUSSION

The surface modification by plasma enhanced chemical vapor deposition on aluminum decreased the mechanical properties.
Bulk SiO_2 resulted in an increase in shear strength, but also induced a decrease in shear energy. All other coatings on aluminum could not surpass the mechanical properties of the reference specimen, although the undercuts provided by the nanoporous layer were promising in theory as the layer was ripped off the metal substrate.
The contact angle measurement shown in Fig. 6 indicates that the adhesion of the coating to the metal failed, because the contact angle of all metal layers is close to the reference. Therefore the PECVD was not suitable for FML.
Sand blasting was the only mechanical treatment that showed an increase in strength, but the shear energy was reduced compared to the reference giving this procedure no advantage. The grinding lowered both interface properties. The mechanical treatment is not as extensive as PECVD but presumably does not inhibit corrosion, therefore sand blasting is unsuitable for FML.
The elastomer interlayer reduced shear strength and increased shear energy greatly. Whether the alteration of properties has a benefit has to be decided by the application. Advantages can be found in the high strain tolerance of the elastomer, which would be desirable under strain induced fractures. The layer is desired to inhibit corrosion and balances the CTE-mismatch. It increased the thickness of the FML, but could suitable for the application in Fiber-Metal-Laminates.

The PECVD coating on steel sheets drastically decreased the mechanical interface properties compared to the reference. Both shear strength and energy were much lower in any configuration of the coating. The contact angle post mortem indi-

cated a failure of the metal/coating adhesion similar to Fig. 6.
Therefore it is concluded that the adhesion of the PECVD-layer to the steel substrate is too low for an application in a laminate. SiO_2 coating may inhibit corrosion, but does not solve the CTE-mismatch.
Mechanical treatment on steel shows promising results, as sand blasting greatly increased shear strength and shear energy. The absolute values of the mechanical properties were the highest, making it an interesting pretreatment. However, the problems caused by contact corrosion and CTE-mismatch cannot likely be solved using mechanical surface treatment. Grinding reduced the interface properties for shear strength and shear energy and therefore is unsuitable.
The elastomer altered the properties on steel similar as on aluminum. It is desired to inhibit corrosion and absorb the CTE-mismatch, making it an interesting choice for the fiber-metal-laminate.
The interface properties of the FML were depicted without focusing on the different densities of aluminum and steel. The values for the steel-based laminates were in the same magnitude as the aluminum-based laminates. Therefore the aluminum-based laminates are more eligible for lightweight applications due to the lower density.

The specimens of all FML modifications failed due to shear stress in the interface between the CFRP and the metal constituent, showing a perfect delamination. No residual CFRP was observed on the metal substrate, proving the desired crack propagation through the interface between laminate partners. Therefore the mechanical properties of the interface between CFRP and metal layer were characterized.
Weidenmann [6] and Hinz [7] forced specimen failure due to shear stress in a different plane namely between the layers of the FRP. The values for shear stress obtained by both were higher than the values in this study.
Hinz [7] monitored fiber/matrix-debonding and matrix cracks in-situ whereas in this study cracks between the laminate partners were observed post mortem. The higher values obtained by Weidenmann [6] and Hinz [7] and the results of this study show that the interlaminar shear strength in the fiber reinforced polymer component is not as critical as the shear strength between the FRP and metal. This conclusion is made based on the crack not propagating through the FRP, but through the interface of the laminate, indicating a higher shear strength in the FRP than in the interface.

7 ACKNOWLEDGEMENT

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