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## Evaluation of surface modified aluminium/polyamide-6 fibre-metal laminates

#### F. Thum<sup>1</sup>, A. Monden<sup>1</sup>, M.G.R. Sause<sup>1</sup>

<sup>1</sup> Institute of Materials Resource Management, University of Augsburg, Augsburg

#### Abstract:

The aim of this study was to investigate the bond strength between thermoplastic carbon fibre reinforced polymers (CFRP) and aluminium alloys with different surface modifications. The materials examined were sandwich laminates consisting of unidirectional carbon fibre reinforced polyamide-6 composites and two different types of aluminium alloys. The configuration chosen for studying the interfacial adhesion is symmetrical with one 0.3 mm thick aluminium foil at the neutral axis of the stack. Various surface modifications like laser structuring, diamond-like carbon (DLC) coating, corundum blasting and different chemical treatments were applied to the aluminium layer. In addition, polyamide-6 interlayers with and without glass-fibres were investigated as an electrochemically insulating layer between aluminium and carbon fibres. The specimens were evaluated using a short beam 3-point bending test to determine the apparent interlaminar shear strength (ILSS). For this purpose, three groups of specimens were stored under standard climate, in a vacuum oven and in hot/wet conditioning for one week, respectively. In addition, potentiodynamic corrosion measurements were performed on the aluminium alloys to evaluate the electrochemical barrier resulting from surface treatments and interlayers. It was observed that 1050 aluminium alloy performed better than 2014 alloy in this aspect. Compared to the configurations without an additional interlayer, interlayers with glass fibre insulation exhibited slightly lower ILSS values with mainly cohesive interfacial failure.

#### **1** Introduction

Due to their specific mechanical properties, Fibre-Metal-Laminates (FML) are increasingly gaining importance with the growing demand for lightweight materials. Compared to conventional fibre reinforced polymers (FRP), the combination of metal and FRP leads to advantages like superior bearing strength, fatigue resistance, transverse fracture toughness and impact resistance [1]. There are different well-established examples for commercial FMLs, like GLARE® ([2], [3]) which is made of glass fibre/epoxy reinforced aluminium alloy or ARALL® [4] (aramid fibre/epoxy reinforced aluminium laminates). We replaced the glass/aramid-fibres with carbon-fibres, expecting to yield higher strength and stiffness. In addition, instead of a thermoset matrix, a thermoplastic polyamide-6 (PA6) matrix was used. This leads to advantages regarding production time and costs. Further, this offers the possibility of thermoforming semi-finished products. However, these modifications result in new challenges regarding the risk of contact corrosion as well as adhesion promotion between aluminium and PA6. Therefore, we studied surface treatments to increase adhesion, protective layers of a polymer (e.g. PA6) or glass-fibre reinforced polymers to prevent contact corrosion. To demonstrate the influence of the surface modifications used in this study, a short-beam shear (SBS) test was chosen to determine the apparent interlaminar shear strength (ILSS) following the conceptual work in [5]. For the evaluation of the electrochemical barrier, potentiodynamic corrosion measurements were conducted on a shortlist of material combinations after the SBS testing.

#### 2 Experimental

#### 2.1 Specimen design and preparation

For the experimental investigations, sandwich plates were produced using a symmetrical layup of eight layers of SIGRAPREG TP C U157-0/NF-T340/46% with a PA6 matrix on two sides of an aluminium foil with a thickness of 0.3 mm. The specimens for the SBS testing procedure of 20 x 10 mm were cut from plates of 100 x 100 mm using a wet cutting table saw. All samples were immediately dried to avoid any water absorption. Due to the different interlayer materials, the resulting thickness ranged from 2.8 - 3.3 mm as shown in Figure 1.

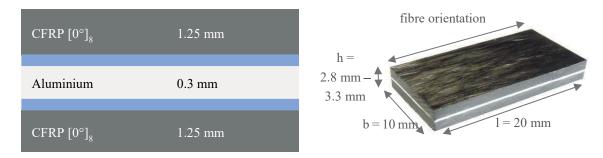


Figure 1: Stacking sequence of the FMLs (left) with schematic interlayers (blue) and specimen dimensions for SBS testing

In this paper, eight combinations of different alloys, surface treatments and interlayers are presented (cf. Table 1). As aluminium alloys, Al 1050A was chosen due to its electrical and mechanical properties and Al 2024 T3 as it is a widely used standard alloy in the aviation industry with good mechanical properties [3].

Laser treatment was used to prepare the metal surfaces for adhesive bonding as well as to clean potentially remaining impurities on the aluminium layer after the cleaning process. The increased surface area and modified topology leads to better mechanical interlocking and increases intermolecular bonding [6]. As a second surface treatment, a silane coupling agent was applied to the aluminium surfaces. On the oxide-surface of the aluminium, the silanes form a layer through covalent bonds [7]. The contact to the CFRP layer (or polymeric interlayer respectively) is improved via chemical bonds which connect to the thermoplastic matrix. The potential difference between aluminium and carbon fibres may lead to galvanic corrosion in case there is direct contact between these materials. To minimize the risk of corrosion, interlayers of PA6 and glass-fibres (GF) with PA6 matrix were added in some cases. As reference, two plates without any surface treatment or interlayers were produced.

No.	1	2	3	4	5	6	7	8
Alloy			Al 1050				Al 2024	
Surface	No	Laser	Silane	Silane	Silane	No	Laser	Silane
treatment								
Inter-	_	_	_	PA6	PA6 +	_	_	_
layer	-	-	-	1 A0	GF	-	_	_

Table 1: Configurations of tested FMLs

Before testing the specimens in SBS, three different conditioning environments were chosen to compare their influence on the FMLs. One part of the samples was conditioned in standard climate for 160 hours, another part was dried in a vacuum oven (T = 80 °C, 160 hours), the third part was stored in a hot-wet environment (T = 80 °C, 95 % humidity, 160 hours). In 3.1, we present the results of standard climate and hot-wet conditioning, as the ILSS values from vacuum drying are similar to the standard climate results.

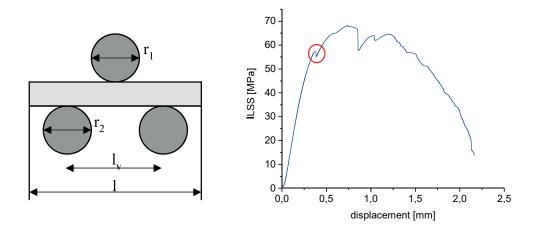
#### 2.2 Methods

#### 2.2.1 Screening via short-beam tests

After conditioning for one week, the FML specimens were investigated via SBS tests following the principles of DIN EN 2563. Load is applied at the centre of the specimen with a given length  $l_v$  between the support which depends on the thickness of the investigated specimen (Figure 2). To evaluate the testing process, the interlaminar shear strength (ILSS)  $\tau$  is calculated from the force at delamination onset  $P_R$ :

$$\tau = \frac{3P_R}{4bh} \tag{1}$$

The maximum shear stress is induced at the specimen's centre plane, where the aluminium layer is located. For thin aluminium layers the ILSS formula gives a reasonable approximation for the actual shear stress at the interfaces between aluminium and CFRP [8]. However, for this study, the calculated values are solely used for comparison between the material configurations, not as actual interlaminar shear strength values. For each configuration, five samples were tested.



**Figure 2:** The short beam shear test is used for comparison of the ILSS values. The first signature within the stress-displacement curve (red circle) indicates the initial failure of the specimen and is used for further evaluation.

#### 2.2.2 Corrosion measurements

The aluminium alloys and the surface modifications were investigated using potentiodynamic polarization. Samples were immersed in a natrium chloride solution (c = 0.5 mol/l) and a scan rate of 5 mV/s was chosen for the measurement using the *Interface 1000* by *Gamry Instruments*. As reference, a saturated Ag/AgCl electrode and as counter electrode a platinum wire was used. The aluminium samples were cut into 10 x 10 mm pieces and cleaned before putting them into the

corrosion measuring setup (Figure 3). The electric current flows between the Platin counter electrode and the sample, which represents the working electrode. The polarization of the sample is varied in the range of -1.2 V to 0 V. For evaluation, the voltage is plotted against the logarithmic current as Evans diagram (Figure 5Figure 5).

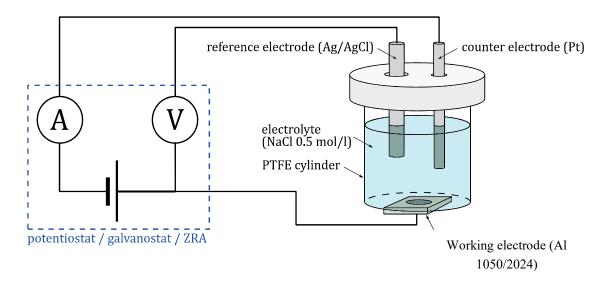


Figure 3: Schematic setup of the potentiodynamic corrosion measurement following [9]

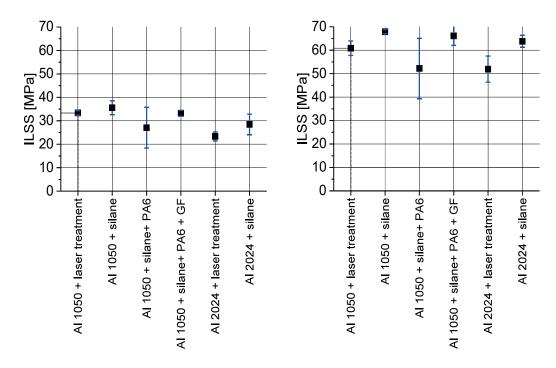
#### **3** Results

#### 3.1 Short-beam shear test

After conditioning in a Hot/Wet climate chamber, the FML samples without any surface modification showed delamination at the interface to the aluminium layer and thus could not be tested. The SBS results presented in Figure 4 show the mean value of five samples for each configuration for the remaining six FMLs.

Two main failure mechanisms could be observed on the SBS samples after testing. For the configurations with silane and additional interlayers, the specimens showed mainly cohesive failure within the CFRP. The samples with laser treatment on the surface showed mainly adhesive failure. The laser treated samples show slightly lower ILSS values when compared to silane treated samples (without additional interlayers). This proves an increased delamination resistance for silane treatment which was also observed in earlier studies with steel/epoxy-based CFRP hybrid laminates [5], [8]. The ILSS values of Al 2024 based configurations consistently lie below the Al 1050 values, especially after Hot/Wet conditioning. This result is corresponding to the better galvanic corrosion resistance of Al 1050.

Despite the use of unreinforced PA6 and GF with lower mechanical properties compared to PA6 based CFRP, these samples in the SBS configuration performed almost as good as the FMLs without additional layers regarding their ILSS values and thus delamination resistance.



**Figure 4:** ILSS values obtained with SBS tests after standard climate conditioning (left) and after Hot/Wet conditioning (right). Five Samples were tested for each configuration.

#### **3.2** Corrosion measurements

Based on the SBS test results, a subset of configurations was examined via potentiodynamic corrosion measurements. Al 2024 was solely used as a reference in two different configurations. The results of the potentiodynamic corrosion measurements of three surface configurations as well as the bare Al 1050 and Al 2024 samples are shown in an Evans diagram (Figure 5), showing the voltage plotted against the logarithmic current. The corrosion potential  $E_{corr}$  and corrosion current  $I_{corr}$  of all configurations are summarized in Table 2.

The polarization curves in Figure 5 show a slight decrease of electrical current for the silane treated Al 2024 specimen throughout the curve while the laser treated Al 1050 specimen stays at almost the same level. However, the  $E_{corr}$  values are in the same region for both alloys as well as the surface treatments. Only the specimen with PA6 interlayers show a clear indication for improved corrosion resistance. Here, the current decreases by several orders of magnitude and  $E_{corr}$  is significantly below the levels of non-treated aluminium samples as well as the ones with surface treatments only.

FML	Al 1050	Al 1050 +	Al 1050 +	Al 1050 +	Al 1050 +	Al 2024	Al 2024 +
		laser treat-	silane treat-	silane treat-	silane treat-		silane treat-
		ment	ment	ment + PA6	ment + PA6		ment
					+ GF		
$E_{corr}$	-0.85	-0.86	-0.8	-0.53	-0.58	-0.79	-0.8
Icorr	1.36x10 <sup>-8</sup>	4.26x10 <sup>-8</sup>	8.17x10 <sup>-9</sup>	2.01x10 <sup>-11</sup>	1.35x10 <sup>-11</sup>	5.8x10 <sup>-8</sup>	2.26x10 <sup>-8</sup>

Table 2: Overview of the extracted corrosion parameters Ecorr and Icorr

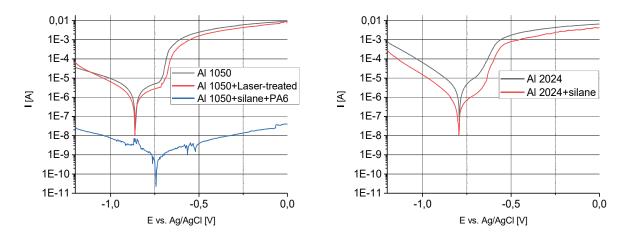


Figure 5: Evans diagrams showing the polarization curves for the differently pre-treated aluminium alloys

The  $E_{corr}$  values for Al 1050 with interlayers of PA6 and GFF are shifted to -0.53 V to -0.58 V compared to the samples without interlayers (surface treatment only), where  $E_{corr}$  is in the region of -0.8 V. In addition, the current rate is significantly below the other samples without interlayers. Therefore, the interlayers decrease the electrochemical potential difference between the carbon fibres and the metal core, which allows to expect a reduced corrosion rate.

#### 4 Conclusions

Eight FML configurations were prepared and conditioned for SBS testing. Furthermore, promising combinations especially with Al 1050 were investigated with a potentiodynamic corrosion testing method.

The surface treatment of both Al 1050 and Al 2024 leads to significantly better performance regarding the interfacial adhesion, which was evaluated using SBS tests to determine ILSS values. In direct comparison, the Al 1050 FMLs show increased adhesion, especially after conditioning the samples in a Hot/Wet environment. FMLs with additional interlayers perform mechanically slightly worse than the ones without interlayers. However, the  $E_{corr}$  values for these samples show good corrosion resistance after potentiodynamic measurement. To investigate the corrosion resistance furthermore, a long term Hot/Wet ageing has been started.

#### 5 Acknowledgement

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