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Strength properties of aluminium/glass-fiber-reinforced laminate with additional epoxy adhesive film interlayer

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Abstract

This paper studies the strength analysis of two types of fibre-metal laminates (FMLs), with a different way of preparing the adhesive coupling between 2024-T3 aluminium alloy sheets and a polymer/fibre layer. In the first variant, the 3M structural adhesive film AF 163-2K is used as an intermediate layer between prepreg and adherends. The adhesive layer is co-cured with prepreg and adherends. In the second FML variant, coupling between adherends was produced using epoxy resin. Two types of strength test, – i.e. tensile/shear test and 90° peel test – were used to measure the adhesion between the layers of FML composite. To measure the joint strength the tensile test was used. The fracture mode and the fracture surfaces were examined and discussed using a scanning electron microscope. The application of the adhesive film as an additional binding agent caused an increase in laminate elasticity. In this way, the strength and fatigue life of the joint increased. The main advantage of adhesive film application was a significant increase in the peel strength of laminate, which reached 289.4%. As a result of this, bonded laminates are very susceptible to normal stresses. However, the application of the additional adhesive film in the analysed 2/1 laminate lay-up increased the mass of the laminate by about 10%. Furthermore, a significant increase in the FML peel strength was achieved, with the increased cost of the additional adhesive film at about 20%.

Keywords: 2024-T3 aluminium alloy, adhesive joints, FML composites, peel strength, shear strength

1. Introduction

Fibre-Metal Laminates (FMLs) are hybrid composite structures composed of thin sheets of metal alloys and layers of fibre-reinforced polymeric materials. FMLs are characterized by enhanced mechanical properties, e.g. high resistance to mechanical fatigue and high strength to density ratio, which is a result of combining the advantages of metallic materials and fibre-reinforced matrix systems [1]. Such laminates are new hybrid materials with properties allowing for impeding and arresting crack growth under cyclic loading.

Fatigue crack growth rates in FMLs are 10 to 100 times slower than those in monolithic aluminium [2]. The most frequently used FMLs are produced based on 2024-T3 or 7075-T6 aluminium alloys and FM94 resin. However, FMLs with high impact resistance can be based on stainless steel and epoxy-based fibre-reinforced prepreg. In order to save weight, magnesium- or titanium-based FMLs were investigated [3]. In addition, high strength, high fracture toughness and high impact resistance of FML offer better safety than aluminium, owing to the high melting point of the reinforcing fibres. The major advantages of metal and fibre-reinforced composites, based on the literature review, are provided by Sinmazçelik et al. [1]. Some types of FMLs, i.e. aramid reinforced aluminium laminates (ARALLs) and glass reinforced aluminium laminates (GLAREs), have been successfully introduced into the Airbus A380, Fokker 27, Boeing 777 and Boeing C-17 [4]. Fibre-metal laminates are 5–10 times more expensive per kilogram than typical aluminium alloys used in the aerospace industry, but they exhibit at least 20% weight savings in the structure [5].

The laminate can be produced by bonding adherends with a prepreg using a press, but the most common process used to produce FMLs involves the use of autoclave processing. Obtaining the very good adhesion of the composite material to the metal substrate is determined by proper surface preparation of the base material (adherend). The lack of adequate adhesion affects the ability of structural discontinuity to occur in the form of porosity, delamination or non-impact, and the lack of full bonding directly affects the quality of the composite materials [6]. A review of adhesively bonded joints in composite materials was done by Banea and da Silva [7], and later updated by Budhe et al. [8]. Adams and Davies [9] paid special attention to new problems which arose when bonding advanced fibrous composites.

The mechanical strength of the FMLs is governed by the adhesion between fibre and matrix, and is the one of the main criteria for its usability in the aerospace industry. The plastic behaviour of laminates is limited to the metal constituents, as fibres do not deform plastically [10]. So, the imposed plastic deformation of the prepreg in the fibre direction is limited by the failure strain of the fibres. Elastic stresses are significant in the laminate when straining occurs in the fibre direction. This results in a rather high springback and/or internal stresses [10]. Owing to many various methods proposed in the literature, the determination of adhesion between adherend and prepreg is not easy. Shear behaviour of composites is a matrix dominated property [5].

The fibre-metal laminate composition may introduce different failure modes [11]: yielding of the metal layers, cracking of the metal layer, fibre failure, and matrix cracking. Failures related to the composition of the FML are the delamination of the composite and metal layers, the delamination of the fibre and matrix, and local buckling of the metal layer.

The purpose of this paper is a comparative strength analysis of two types of fibre-metal laminates, with a different way of preparing the adhesive coupling between 2024-T3 aluminium alloy sheets and glass/epoxy prepreg. In the first type of FML, coupling between adherends was produced using epoxy resin. In the second type of FML, the additional adhesive film was applied between prepreg and adherends to improve the adhesive strength between the metal and the Glass-Fibre-Reinforced Polymer (GFRP) layer. Three types of specimens were prepared to study the strength of FMLs using the tensile test, tensile/shear test and 90° peel test.

2. Experimental procedure

2.1. Adherend

2024-T3 precipitation hardenable aluminium alloy sheets with a thickness of 2 and 0.4 mm were used in this study for the fabrication of FMLs. This aluminium was selected because it is widely used to manufacture adhesively bonded aircraft structures, owing to its excellent specific strength and fatigue performance, good conformability and surface finishing capabilities. The mechanical properties of the used aluminium alloy sheets determined in the uniaxial tensile test according to ISO 6892-1 standard [12], are listed in Table 1. Five specimens were tested and average values of mechanical parameters value were determined. The confidence limits for 95% confidence intervals are listed in brackets in Table 1.

The tensile properties of the test specimens with rectangular cross section depend on the specimen thickness. This effect is discussed in the literature and in the case of Al alloys, Suh et al. [13] has shown that the increasing of specimen thickness leads to an increasing influence of the free surface on the flow stress of grains located at the specimen surface. Furthermore, the strain hardening phenomenon, expressed by the relationship of true stress and true strain, varies with specimen thickness. This may be attributed to the variation of strain paths due to geometrical size effects [14].

Table 1. Basic mechanical parameters of 2024-T3 aluminium alloy sheet

Thickness t, mm	Young's modulus E, GPa	Poisson's ratio ν	Yield stress $R_{p0,2}$, MPa	Ultimate tensile stress R_m, MPa
0.4	72.87(0.66)	0.33 (±0.006)	302 (±0.25)	449 (±0.94)
2	70.75 (±1.75)	0.33 (±0.007)	336 (±1.64)	478 (±0.72)

The aluminium alloy surface treatment prior to bonding is critical to ensure good adhesion between the metal and the adhesive. Degreasing is the minimum pretreatment that is usually carried out prior to bonding [1]. Anodizing produces a thin oxide film to avoid or inhibit the corrosion event [15]. The adherends' surfaces were anodized under the following procedure. The oxide coatings were produced onto the 2024-T3 substrate in the anodizing process. The specimens were abraded with a sand paper grade 320, rinsed with water and degreased in the NaOH aqueous solution (100 g·dm^{-3}) for 1 minute at 25°C, rinsed with deionised water, and pickled in the HNO_3 aqueous solution (400 g·dm^{-3}) for 1 minute at 25°C. Subsequently, they were anodized in the H_2SO_4 aqueous solution (300 g·dm^{-3}) at 15°C. The constant current density equal to 1 A·dm^{-2} was applied. For a substrate thickness equal to 2 mm, anodising time was equal to 25 minutes and 10 μm thick coatings were produced. For thinner substrates (0.4 mm) the anodizing time was equal to 10 minutes, and thinner coatings were produced (6 μm). When the anodizing process was completed the coatings obtained were rinsed with deionized water and dried in air. Their thickness was determined using the eddy-current method (Dualscope FMP100, Fischer).

Prior to bonding, the aluminium surfaces were primed with EC-3924B (3M™, Maplewood, Minnesota, USA). 3M™ Scotch-Weld™ Structural Adhesive Primer EC-3924B is a sprayable or brushable corrosion inhibiting adhesive primer. It provides a high degree of protection against corrosive environments both inside and outside the bond line. The description of this primer, according to the manufacturer's data, is as follows: base - synthetic resin, density 0.887 kg/dm^3, flash point -14.4°C, consistency - thin liquid, solids content - 6 ±1.0%.

2.2. Surface roughness

Surface topographies of the sheets were measured before primer application with a Talysurf CCI Lite 3D optical measurement system equipped with objective 5x. Parameters of surface textures were calculated using TalyMap software. Each profile has an area of 3.3 × 3.3 mm with a resolution of 1024 × 1024 points. Textures of surfaces were only levelled and digital filtration was not used. The surface topography of the sheets used after anodizing process is presented in Fig. 1.

The roughness average *Sa, and* the 10-point peak-valley surface roughness *Sz* were measured in five different areas on each surface. The average of the five measured values was considered as the average surface roughness for each specimen. For a sheet thickness of 0.4 mm the following roughness parameters were obtained: *Sz* = 29.4 (±1.39) µm, *Sa* = 1.97 (±0.13) µm, whereas for the sheet with a thickness of 2 mm: *Sz* = 37.1 (±1.26) µm, *Sa* = 1.79 (±0.07) µm. The confidence limits for 95% confidence intervals are listed in brackets.

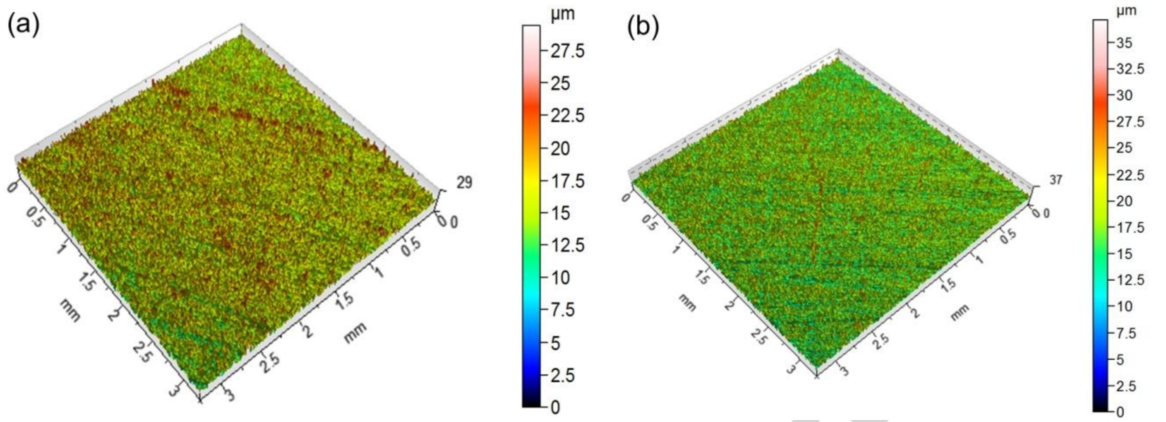

Fig. 1. Surface topography of sheets after anodizing process for sheets with thicknesses of (a) 0.4 mm and (b) 2 mm

2.3. Specimen preparation

Two types of FMLs consisting of a three-plie lay-up were fabricated in this research. In the first configuration of FML (Fig. 2a), 3M Scotch-Weld^{TM} AF-163-2K thermosetting modified epoxy adhesive film (3M, St. Paul, Minnesota, USA) was used as an intermediate layer between glass/epoxy woven HEXPLY-916G (Hexcel Corporation, Stamford, Connecticut, USA) prepreg and adherends. A laminate is symmetric, so the plies above the mid-plane are a mirror image of those below the mid-plane. In the second type of FML (Fig. 2b), the laminate was prepared without adhesive film between the glass/epoxy matrix and adherends.

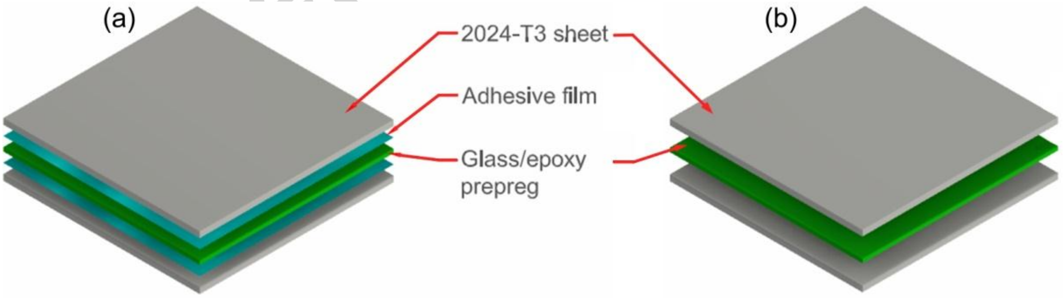

Fig. 2. Stacking configurations of FML (2/1 lay-up) variants considered: (a) with adhesive film; (b) without adhesive film

The laminates were autoclave-cured under the following conditions: heating speed 2^{o}C/min, curing temperature 135°C, pressure in autoclave chamber during curing 3 bar, curing time 90 min., and cooling speed after curing 3^{o}C/min. The negative pressure of -0.7 bar in the vacuum bag is maintained until the curing temperature is reached, which takes 30 minutes. The cure cycle of the analysed FMLs is presented in Fig. 3.

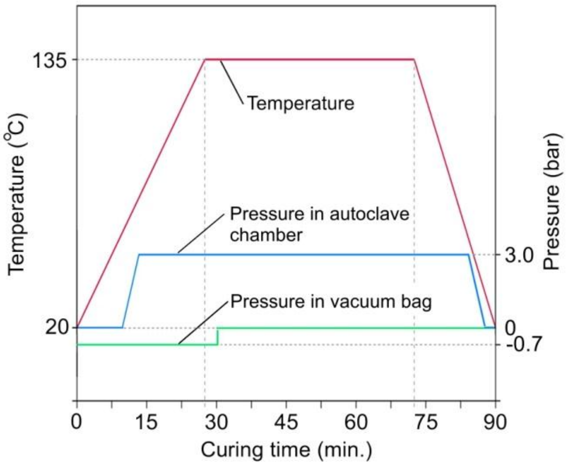

Fig. 3. Autoclave cure cycle for FMLs studied

The test composites were made in the form of panels with the following dimensions: for tensile testing – 250 mm × 360 mm, for tensile/shear testing – 200 mm × 360 mm and for peel test - 300 mm × 360 mm. After panel curing, for the strength tests the samples were cut using a high-pressure water jet technique. The treatment was carried out at a water pressure of p = 350 MPa, an abrasive mass flow rate of 300 g·min^{-1}, and transverse speed v_f = 250 mm·min^{-1}. The rest parameters of the abrasive water jet cutter were as follows: water orifice diameter d = 0.35 mm, mixing nozzle diameter D = 1 mm, grade of abrasive garnet #80.”

2.4. Tensile test

The dimensions of the samples and the experimental procedure for uniaxial tensile testing were based on ASTM standard D3039 [16]. The dimensions of the samples for the variant of a 2/1 lay-up are presented in Fig. 4a. Testing was carried out using universal testing machine Zwick/Roell Z100, with maximum capacity of 100 kN. The testing speed was 5 mm/min. Tensile tests were performed at room temperature and five specimens for each FML variant were tested to evaluate the average tensile strength of the laminate.

2.5. Tensile/shear test

The conditions of static tensile/shear test were assumed based on ASTM standard D1002 [17], which was developed for the determination of apparent shear strength of single-lap-joint adhesively bonded metal specimens. The single-lap samples consisted of two sheets with a thickness of 2 mm, and between them the laminate was adhesively bonded. Prior to cutting, the panel with dimensions of 360 × 200 mm was milled to obtain a lap on both sides of the laminate, with a width of 12.5 mm (Fig. 4b). Next the panel was cut with a water jet to prepare samples with a width of 25 mm (Fig. 4b). Tensile/shear tests were performed at room temperature and five specimens for each FML variant were tested to evaluate the average shear strength of the laminate.

2.6. 90° peel test

To investigate the peel strength of the laminates the 90° peel test was carried out according to DTD 5577 [18]. The tests were carried out using universal testing machine Tinius Olsen H25K-T. A 2 mm thick 2024-T3 aluminium alloy sheet (rigid adherend) was adhesively bonded to a 0.4 mm thick 2024-T3 aluminium alloy (flexible adherend), at a distance of 200 mm. The width of the samples was 25 mm (Fig. 4c). The average values of the peel strength were evaluated based on the ten tests for both analysed FML variants.

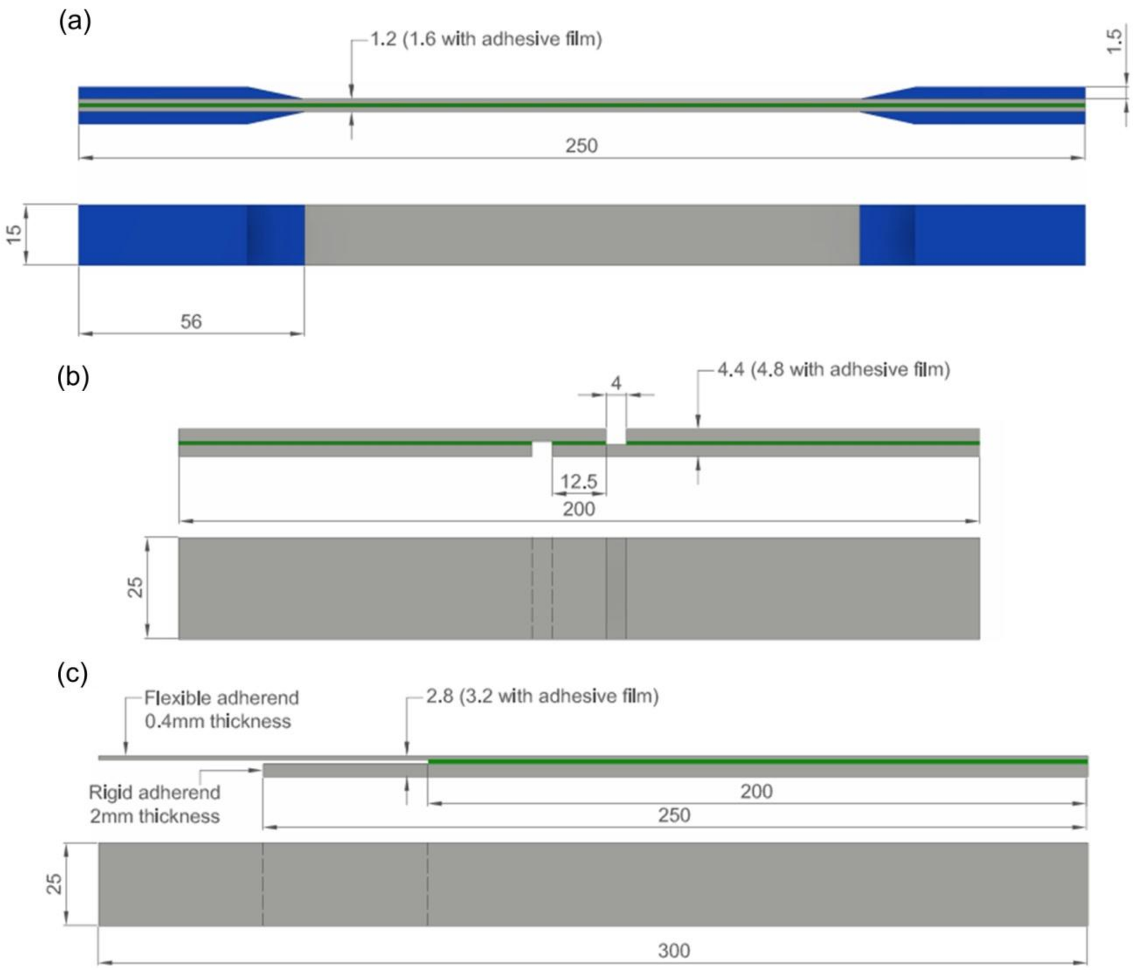

Fig. 4. Geometry of FML test specimens for: (a) tensile test; (b) shear strength test; and (c) 90° peel test (dimensions in mm)

2.7. Fracture surface analysis

The morphology of the specimens after the experiments and the chemical composition of their surfaces were determined using scanning electron microscope (SEM) HITACHI S-3400N with an energy-dispersive spectroscopy (EDS) and a wavelength-dispersive spectroscopy (WDS) systems.

3. Results and discussion

3.1. Visual analysis of FMLs

Individual variants of the samples were subjected to optical analysis to illustrate differences in the cross-sectional view of the FML without adhesive film (Fig. 5a) and with adhesive film (Fig. 5b). In the case of a variant with adhesive film, the thickness of the polymer-fibre composite layer is about two-fold higher than in the case of the variant without adhesive film. The final adhesive thickness was determined by substracting the adherend’s thickness from the total thickness. It is also noticeable that in the autoclave-curing process, the epoxy resin, as the saturant of the prepreg, was mixed with the adhesive film-based resin. Budhe et al. [8] stated that in the co-curing process involving woven fabrics, a uniform adhesive thickness was difficult to achieve due to the geometry of the fabric itself, resulting in significant differences in the thickness.

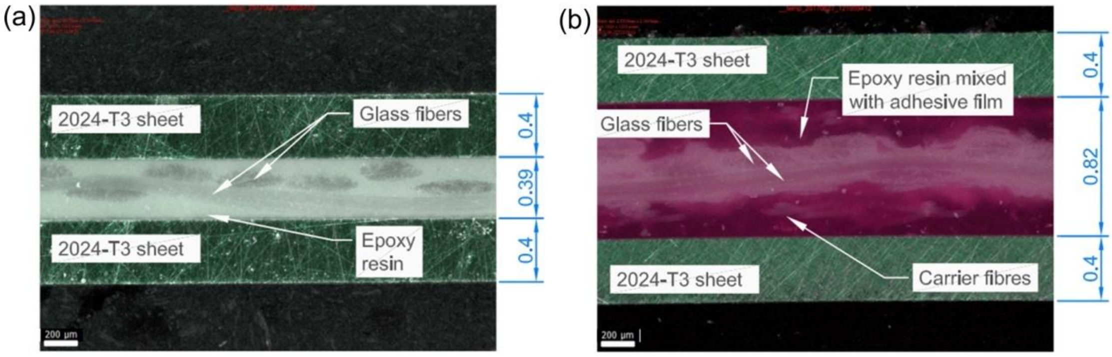

Fig. 5. Cross-sectional views of composites studied: (a) without adhesive film; (b) with adhesive film (dimensions in mm)

3. 2. Tensile test

Stress-strain relations for both variants of the samples are presented in Fig. 6. A significantly lower tensile stress value for the FML fabricated with an additional adhesive film is due to an increase in the composite thickness of about of 33%, by using two layers of the adhesive film. However, the adhesive film used only contributes slightly to an increase in the composite weight; namely, 9.8%. As a consequence, the specific strength, also known as the strength-to-density ratio, was determined on the basis of the strength values for analysed FML variants. The specific strength of the composite without the adhesive film was 152.8 kN·m/kg, and for the variant without the adhesive film 142.0 kN·m/kg. The strain to failure is 8.5% lower for the variant with the adhesive film (Fig. 6). If it is assumed that the strain to failure is a measure of ductility, Gonzales-Canche et al. [19] concluded that the FML exhibits an improved ductile behaviour when compared to its constituent materials. Table 2 summarizes the basic results of the tensile strength test and the mass of samples.

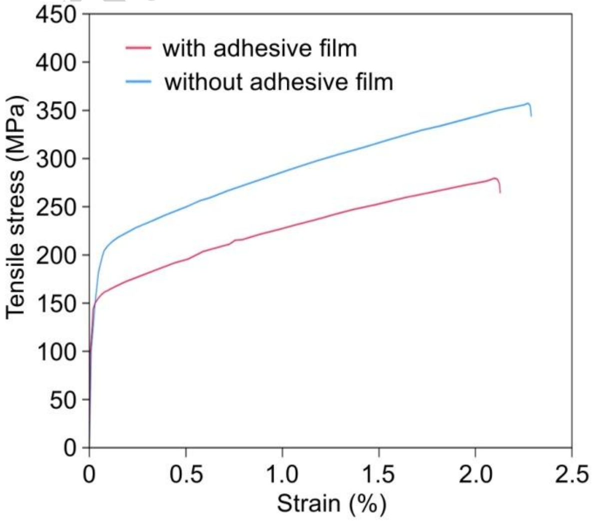

Fig. 6. Typical stress-strain curves of tensile test of FML composites studied

Table 2. Mechanical properties of specimens

Parameter	FML without adhesive film*	FML with adhesive film*
Mass (g)	10.623 (±0.095)	11.663 (±0.113)
Tensile strength (MPa)	360.729 (±4.76)	267.60 (±3.05)
Specific strength (kN·m/kg)	152.815 (±3.35)	142.042 (±1.46)

* - The confidence limits for 95% confidence intervals are listed in brackets.

The difference in Young's modulus for the aluminium alloy sheet and hardened prepreg means that in the uniaxial tensile test, local shear-stress damage of the adhesive layer between the adherends and prepreg occurs prior to sample destruction. This effect occurs in both variants of the composite. Fig. 7 shows the SEM images of the broken composite structures fabricated without the adhesive film. The difference in the mechanical properties of the adherends and the glass/epoxy matrix mean that tensile specimens are prone to the detachment of layers from each other, which leads to failure. The top and bottom light layers (Fig. 7) indicate that the structure of the aluminium sheared owing to the application of tensile load. The middle layer shows the deformed fibre-reinforced structure. The application of tensile load also causes shearing of the fibres, so in the magnifications of regions 1 and 2 (Fig. 7) groups of protruded fibres are visible on the fracture surface. In the magnifications of region 1 and 2 (Fig. 7), there are also visible groups of fibre fractured almost at 90° to the loading direction. This effect was also observed by Rajkumar et al. [20], and is due to good bonding strength between epoxy matrix material and carbon fibres. In GFRP, the debonding between pullout fibres and the matrix becomes easier than in Carbon Fibre-Reinforced Polymers (CFRP) [20]. The damage mechanism in metal-fibre laminates is a combination of matrix cracking, delamination, fibre shear out/ pull out and fibre fracture (Fig. 8). In the tensile test excellent adhesion between plies plays an important role in enabling an increase in the levels of global plastic deformation observed in the aluminium sheets, delaying the formation of localized strain that leads to cracking [19].

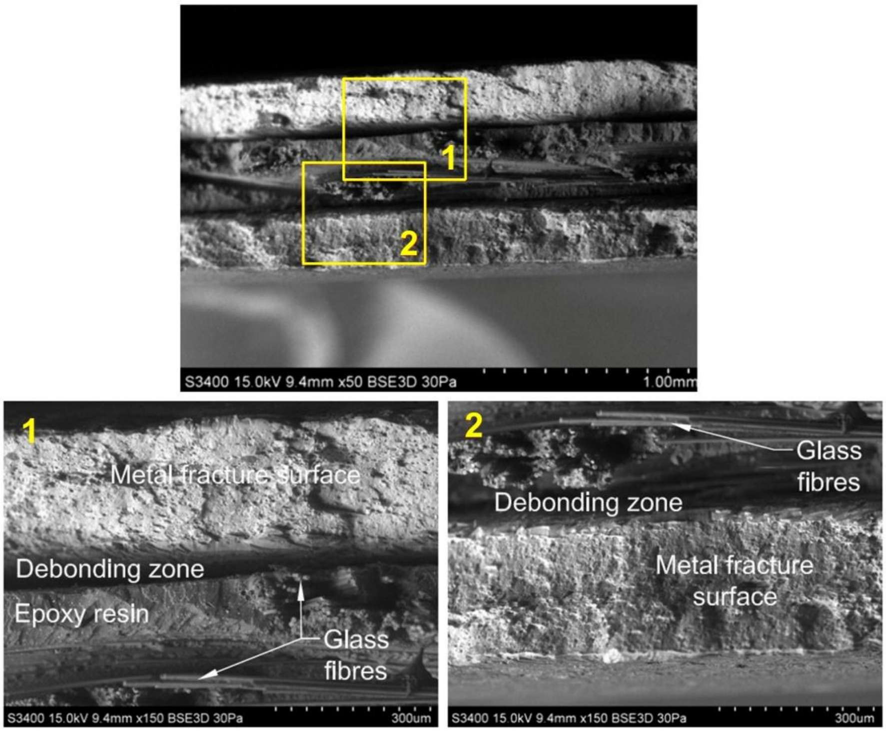

Fig. 7. SEM micrographs of fracture surface of specimen without adhesive film after the tensile test

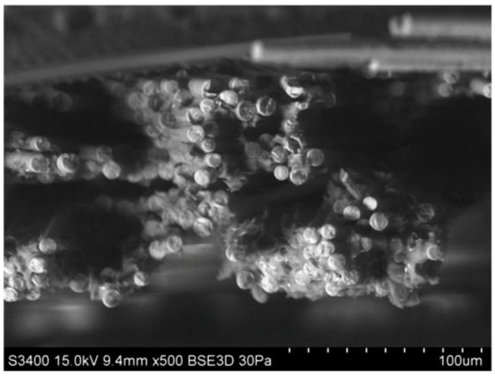

Fig. 8. Glass fibres fracture in GFRP layer after the tensile test

3.3. Tensile/shear test

Shear behaviour of FMLs is a dominant matrix property. The results of tensile/shear tests showed similar shear-stress values for both analysed FML variants. In the variant with the adhesive film, the average value of shear strength was 6.52 MPa, whereas for the variant without the adhesive film it was 6.74 MPa. The confidence limits for 95% confidence intervals for the variant with the adhesive film and the variant without the adhesive film were ±0.46 and ±0.36, respectively. The shear strengths for both FML variants are statistically similar. However, the FML with the adhesive film exhibits longer displacement prior to fracture (Fig. 9). So, the addition of the adhesive film as an additional bond between plies results in a slight increase in the elasticity of the composite. The increase in displacement prior to fracture for the variant with adhesive film was equal of 11.3% (±2.954).

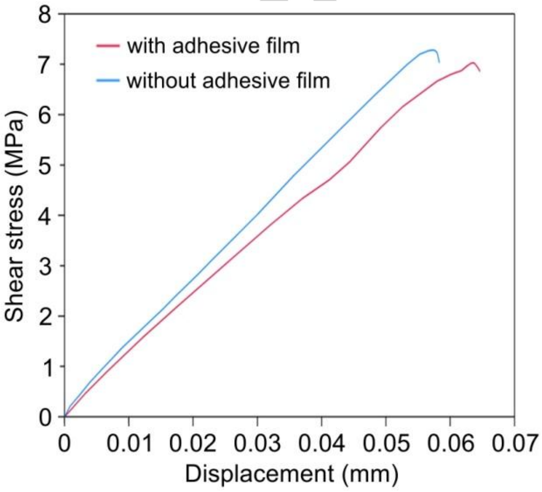

Fig. 9. Typical tensile/shear test curves for FML composites studied

By analysing the fracture surfaces at the macroscale, in both variants of laminate the apparent adhesive failure was observed between the adhesive and one of the adherends. The SEM images of individual fracture surface show the noticeable differences between variants. In the case of the variant without an adhesive film (Fig. 10a), the residue of the epoxy resin is visible on the adherend surface (1 in Fig. 10a), but only near the edges of the bonded surface. The remaining part of the adherend surface is free from clear traces of resin. This may be due to the effect, at the initial stage of the destruction, of the normal stress component at the edge of the joint, in which a small fragment could lead to tearing off the bonded layers. However, the remaining part of the bonded surface undergoes classical shear in the layer of epoxy resin bonding to the adherend, which leads to adhesion failure. For the samples with an adhesive film layer (Fig. 10b), in the microscope magnification it was noted that the adhesive residues regularly covered the whole surface of the adherend.

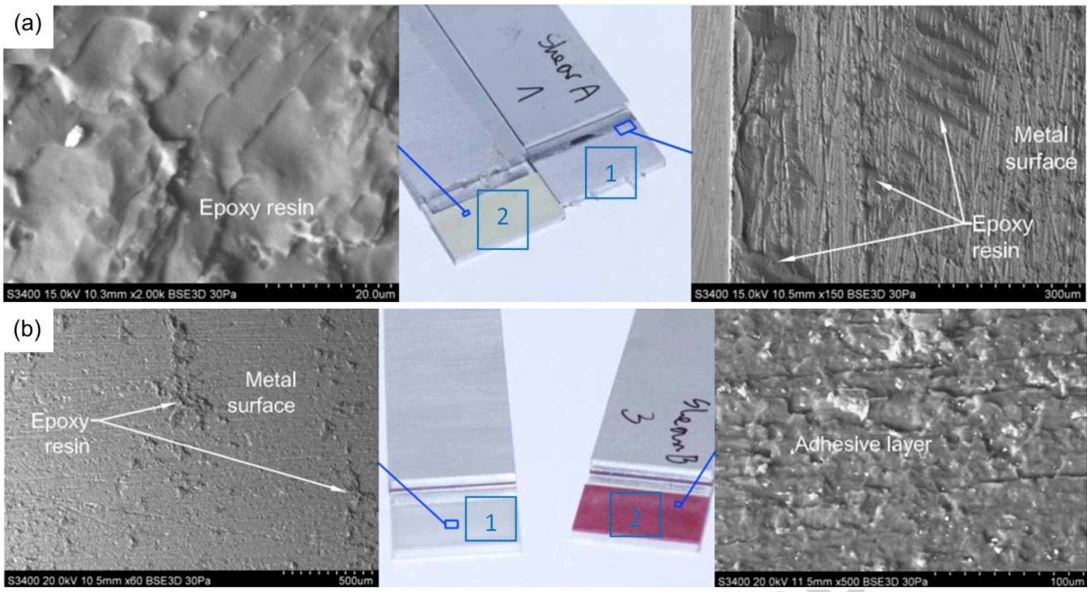

Fig. 10. SEM micrographs of fracture surface after tensile/shear test: (a) specimen without adhesive film; (b) variant with adhesive film, on 1. adherend surface and 2. GFRP surface

3.4. Peel strength test

FML variants with the same configuration of adherends were tested in peel tests. In both cases the thinner adherend („flexible" according to the Fig. 4c) is pulled from the substrate. The results of the 90° peel tests showed significant differences in the strength of the considered variants of FMLs. Fig. 11 presents an example of load variation during the 90° peel test. With normal stress applied to the adhesively bonded layer, the use of the adhesive film as an additional binder resulted in a significant increase in peel load. The peel strength is calculated during a peel test at a constant speed rate divided by the average force required during the test by the unit width of the samples.

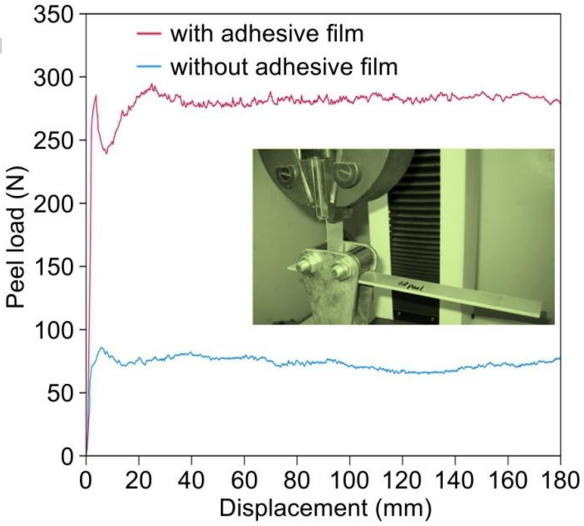

Fig. 11. Example of load-displacement graphs measured during peel tests

For a laminate variant without the adhesive film, the peel strength was 2.83 N/mm, and in the variant with the adhesive, the strength was 11.02 N/mm. The indicated that the difference between both analysed laminate variants translates into a significant increase in the laminate quality. In the adhesively bonded structures the normal stress is very dangerous in terms of joint strength. So, any method that leads to an increase in the peel strength of the FMLs is very desirable.

For laminates without an adhesive film (Fig. 12a), adhesion failure was observed. On the other hand, each of the samples with an additional adhesive film (Fig. 12b) was characterized by cohesive failure along the complete debonding length, which confirms that the adherends are properly bonded and that this bond will endure. De Freitas and Sinke [21] found that in the peel test the fracture mechanism of a cohesive failure was independent of the peeling-off adherend (aluminium or composite). It is typical [i.e., 22] that there are no significant changes in either the failure mechanism along the debonding length of the specimen, or the peel load along the displacement (Fig. 11). Small residuals of epoxy resin on some areas of the adherend surface are noticed based on the SEM image (Fig. 12a) of a variant without the adhesive film. This means that cohesive failure has occurred. The increase in the percentage of cohesive failure areas is the reason for an increase in the peel load [22].

Peel energy is commonly used to describe the adhesion characteristics of the facesheet and core material. The peel energy (per unit area) is dissipated by all the energy-dissipating processes involved in the broad area associated with the peel front. In most practical circumstances, the magnitude of peel energy is largely determined by the extent of dissipation within the materials of the adhesive bond [23].

The increase in average peel strength at about 289.4%, from 2.83 N/mm to 11.02 N/mm, can be attributed mainly to the type of failure. De Freitas and Sinke [22] indicated that adhesive failure leads to lower peel loads than cohesive failure, as expected. The peel force includes the force required to deform flexible adherend plastically and the decohesive force of the interface [22]. However, the contribution of the interface adhesion of the thin film to the peel load is in the order of 100 times lower than the plastic deformation of the thin film [22]. In the case of the laminate with the adhesive film (Fig. 12b), micrometric surfaces with an adhesive type of failure are observed. In the fracture surface, fragments with the carrier fibre are also revealed. The carrier is used to control bondline thickness and adhesive bleeding during the curing process [8].

Peel stresses are very dangerous considering the delamination of the FML plies. There are some methods to reduce peel stresses in adhesive joints with composites. For example, if thermal stresses are not important, da Silva and Adams [24] increased the joint strength by using an internal taper and adhesive fillet arrangement.

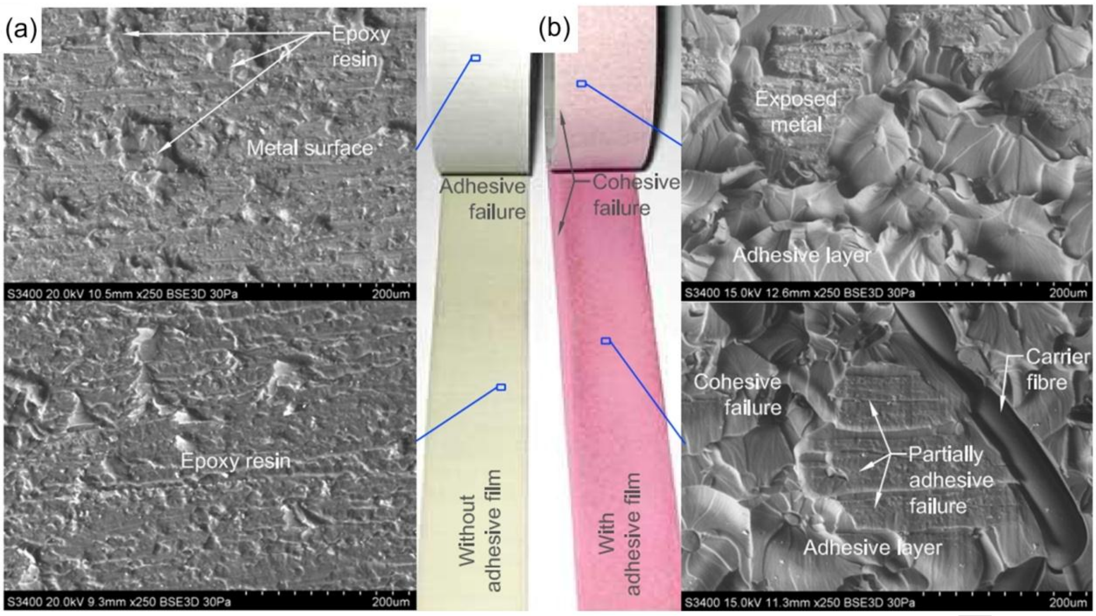

Fig. 12. SEM micrographs of fracture surface after peel tests of specimens (a) without and (b) with adhesive film

4. Summary and conclusions

The results of the strength tests indicate both the positive and negative effects of using an additional adhesive film to bonding the individual plies of the metal-fibre composite.

The main advantage of using an adhesive film between adherend and prepreg is a significant increase in the peel strength of the FML, which was 289.4%. This is an important feature of metal-fibre composites. Although the adhesively bonded structural joints should predominantly transmit shear stresses, in practice, this type of joint is often subjected to normal stresses that cause peeling or tearing. Hence, the application of an adhesive film as an additional binder may have a decisive influence on the strength of the layered structure.

A certain advantage of the laminate with the adhesive film may be a slight increase in the flexibility of the joint, which hypothetically may prove to have a beneficial effect in the case of cyclic loads. Increased elasticity of the joint may contribute to the improvement of the strength and durability of the composite. Considering the weight of the laminate, the application of two additional adhesive films, in the fabricated variant of the FML with lay-up 2/1, results in an increase in the composite weight of about 10%. The composite thickness is increased by about 30%. Finally, the economic factor should be noted: the double layer of the adhesive film per one layer of prepreg increases the cost of materials by approximately 20%.

However, in some cases increased peel strength of the FML equal to about 290%, can be more important than the cost of FLM fabrication. The analysed laminate with the glass fibre/epoxy matrix and the adhesive film is a combination of extended elasticity and high peel strength from the composite layer, resulting in great performance for aircraft applications.

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