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Influence of anodization of aluminum 2024 T3 for application in aluminum/Cf/ epoxy laminate

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ABSTRACT

This work deals with the processing and characterization of aluminum/carbon fiber/epoxy composites obtained from anodized and non-anodized aluminum plates. In this work, the electrochemical treatment employed was the phosphoric anodization, after chemical stripping. After this treatment, the fiber metal laminate (FML) coupons were laminated, the aluminum plates were interleaved with CF/epoxy prepreg layers. The next step was the use of hot compression molding technique in order to prepare the FML with and without anodized aluminum surface treatment, for comparison purposes. Several characterization techniques were used, such as: impact and perforation resistance analysis, laminar, translaminar (Iosipescu) and compression shear strength, in addition to optical macroscopy. After the analysis of the results, it was verified that the electrochemical treatment slightly reduces the impact and the perforation resistance of FML studied, because a more adhered interface promotes larger internal tensions and consequently reduces the capacity to absorb energy until failure. However, the adhesion properties were shown to be superior in the composites produced with the anodized plates. Additionally, the treatment proved to be an important means of obtaining FMLs, since most of the plates produced with non-anodized aluminum delaminated after a few weeks in stock, while the FML obtained from anodized aluminum did not delaminate after months of their production.

1. Introduction

In order to reduce the specific mass and at the same time, to present good mechanical properties, the fiber metal laminate (FML) composites were developed (late 70s). This material was presented as one of the most promising technologies in the aeronautical market. High mechanical properties, which overcome the properties of its individual constituents, make the laminated composites an important milestone in the aeronautical application, where access to new and more efficient technologies is fundamental for a good performance, being the ARALL (aramid reinforced aluminum laminate), GLARE (glass aluminum reinforced) and CARALL (carbon aluminum laminate) the most important representants of this material [\[1](#page-10-0),[2](#page-10-0)].

When the structure of the aircraft has a lower specific mass at the same time at high properties, the overall performance of the aircraft tends to improve due mainly to the reduction in fuel consumption. However, the use of FML composites still needs to be studied in order to understand better their limitations, because when in service, these materials can suffer a process of progressive damage by different failure mechanisms [[3](#page-10-0)], such as: fiber tension, compression damage, matrix tension and compression damage and inter-laminar delamination damage [[19](#page-10-0)].

Damage to FML composites generally occurs due to internal stresses between the composite layers or between the reinforcing fibers and the matrix, the most common damages are breaking of the matrix or fibers and delaminations [\[3\]](#page-10-0). The delaminations are detachments between two adjacent layers so this kind of damage is limited to a plane.

In order to obtain good mechanical properties, the composite layers must have good adhesion to each other. The adhesion between two surfaces arises from attractive short-range forces between atoms on each surface. In the case of a metal/fiber laminate, this adhesion strength depends on how the polymer interacts with the metal [\[4\]](#page-10-0).

One way to increase adhesion at the interface is the electrochemical treatment (anodization) of the metal plates that constitute the hybrid composite. Such treatment increases the surface area (increase in roughness) and increases the number of polar groups on the surface of

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Fig. 1. (a) the perforator used, (b) the sample positioned in the sample holder and (c) the perforator after impacting the sample.

the metal. These modifications increase the mechanical and chemical adhesion at the metal/composite interface [[5](#page-10-0)].

Thus, this work involves the processing of FML composites constituted of epoxy, carbon fibers and anodized 2024 T3 aluminum alloy. The phosphoric acid anodization of aluminum was carried out, aiming at an improvement of the interface of the final product. The composites were characterized by perforation, impact, compression shear, interlaminar shear and Iosipescu tests. After the mechanical tests the samples were submitted to optical macroscopy in order to evaluate the extent of the damage generated.

In view of the above, this work has as main objective to evaluate the influence of the electrochemical treatment (of the aluminum plates used in the composite manufacture) on the impact resistance and shear strength of Al/CF/Epoxy laminates [[5](#page-10-0)].

2. Experimental

2.1. Materials

The aluminum alloy used for FML lamination was the 2024 - T3 aluminum, purchased from Alto Parts, SP, Brazil, in the form of 0.4 mm thick aluminum plates. Carbon fiber/epoxy prepreg was prepared using a carbon fiber plain weave fabric supplied by Hexcel Company, USA and epoxy resin. The silicone-based release agent (Polidesmo®) was used to prevent adhesion of the material to the press plates.

2.2. Anodization of 2024 - T3 aluminum alloy

The anodized aluminum plates were previously cut in accordance with the specific standard of each test with the addition of 20 mm, to better couple the plates during the anodizing process. The plates were washed with water and commercial detergent then dried with a dryer at room temperature. They were then cleaned with acetone to ensure maximum removal of dirt and grease from the plates. The anodization process was based in previous works [[20,21](#page-10-0)].

Before being anodized, the plates underwent a chemical pickling process. This process is important to ensure a good anodization result, as it helps in the superficial uniformity of the plates, leaving a more propitious surface for the anodization. In this process, the plates are placed in an extremely basic medium for oxidation and then placed in an acidic medium for removal of the surface oxide.

The basic solution used was a 10% NaOH w/w. The chemical stripping process was carried out hot (about 60 \degree C), each plate was immersed in this bath for 60 s. After chemical cleaning, the plates were immersed into 85% nitric acid (HNO₃) w/w, for 90 s, at room temperature. After neutralization of the base with the acid medium, the plates were rinsed thoroughly with running water and then with distilled water. Finally, they were dried with a drier.

Direct current anodization was performed in a three-liter glass Becker, sufficient to accommodate the aluminum plates and the counter electrodes (copper). A 12% w/w phosphoric acid solution (H_3PO_4) , a power source CC Agilent model E3634A (25 V-7 A), a thermostat (used to ensure bath temperature stability) and a bubbler (used to maintain the stirring of the bath) were employed.

Before starting the treatment, the thermostat was switched on, to ensure that the initial bath temperature was always constant (approx. 23 °C), the ideal temperature for the procedure $[20,21]$ $[20,21]$ $[20,21]$. The plates were then anodized for 10 min under a voltage of 10 V. The stabilization of the electric current occurred around 2.7 A to produce a laya approximately 0.5 μm [\[20,21](#page-10-0)]. After the anodization the plates were washed thoroughly in running water and then with distilled water. The plates were then oven dried for 30 min at 60 °C.

2.3. Processing of aluminum/CF/epoxy composites

The carbon fiber/epoxy prepreg was cut into the dimensions of the aluminum plates. For each laminate, three aluminum plates interlayer with two composite agglomerates were used (each agglomerate was composed of 4 prepreg layers).

With the stacked materials, the laminate was submitted to a hot molding process in a Carver press, model CMG100H-15-X, at a temperature of 120 �C and pressure of 0.6 MPa for 180 min, in order to cure the epoxy resin. The press plates were previously lubricated with the Polydesmo release agent.

On each plate the specified dimensions of the specimens were marked. The cut was performed on a conventional band saw. After cutting, the test specimens had their sides subjected to a sanding process for better finishing and dimensional accuracy with sanding granules nº 100. Finally, the dimensions of the specimens were measured for subsequent calculation of the tensions in each characterization.

2.4. Perforation test

The perforation test was carried out with a drop tower for impact tests up to 3 kJ. This test was based on DIN EN ISO 6603-2 (Plastics - Determination of puncture impact behavior of rigid plastics). A metal indenter with a diameter of 20 mm and an impact energy of 207 J (corresponds to an impact velocity of 4.5 m/s) was released into the

Fig. 2. (a) setup used in the impact test and (b) the sample placed in the sample holder.

composite samples. The response of the material was characterized by the maximum force (F_{max}) , the displacement, in which the force has dropped to half its maximum value (l_p) and the absorbed energy (AE_p) . The total mass of the punch was 20.7 kg. Seven samples without treatment and 7 treated samples of 70 mm \times 70 mm were used. [Fig. 1](#page-1-0) shows (a) the perforator used, (b) the sample positioned in the sample holder and (c) the perforator after impacting the sample.

2.5. Impact test

The impact test was carried out using a drop tower for impact tests up to 3 kJ. The test was based on E DIN EN 6038: 2014-08 standard (the same standard AITM 1-0010 from Airbus). Six untreated and six treated samples of 150 mm \times 100 mm were used. The selected impact energies were 9 J, 12 J, 16 J, 25 J, 30 J and 40 J. Fig. 2 shows (a) the setup used and (b) the sample positioned in the sample holder.

2.6. Optical profilometry

The optical profilometry characterization was performed in the impacted samples to evaluate the size of the damage caused in the composite plates, considering the area of 50 mm \times 50 mm as analyzed region. This test was carried out using the FRT profilometer MicroProf of the Fries Research & Technology GmbH, whose lateral resolution is 1 μm and the vertical resolution is 3 nm.

2.7. Compression shear test (CST)

The CST test was performed on 6 samples of the treated Al/Epoxy/CF composite and 6 of the untreated Al/Epoxy/CF composite. The equipment used was a Shimadzu universal test machine, the speed used was 0.25 mm/min and the load cell used was 5 kN. In this test, a device coupled to the universal testing machine is used, to allow the evaluation of the shear strength of a test specimen when subjected to a compression force. The device is equipped with two lateral arms (one movable and one fixed) that accurately adjust the position of the sample (10 mm \times 10 mm) so that the stress is applied in the direction perpendicular to the shear plane of the specimen [\[6,7](#page-10-0)].

2.8. Interlaminar shear test (ILSS)

The ILSS test was performed on 12 samples of the treated Al/Epoxy/ CF composite and 12 of the untreated Al/Epoxy/CF composite. The

Fig. 3. Scheme of the position of the microscopic sample (red) that was removed from the impacted plate. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

equipment used was a Shimadzu universal test machine, the speed used was 0.5 mm/min and the load cell used was 5 kN, following the ASTM [D2344](astm:D2344) standard.

2.9. Iosipescu test

The Iosipescu shear test was performed on 7 samples of the treated Al/Epoxy/CF composite and 7 of the untreated Al/Epoxy/CF composite. The equipment used was the Shimadzu universal test machine, the speed used was 0.5 mm/min and the load cell used was 5 kN, following the ASTM 5379M/05 standard.

2.10. Optical microscopy and stereoscopy

The impacted plate, the CST and the ILSS samples were evaluated by optical microscopy to evaluate the damage caused by the mechanical tests. Samples from the impacted plate (Fig. 3) and the specimens of ILSS and CST were embedded in resin (room temperature) and sanded to sandpaper nº1500. The microscope used was the Nikon EPIPHOT 200. The samples tested by Iosipescu did not undergo previous preparation and were evaluated by Stereoscopy. The stereoscope used was the Stemi 2000 from Zeiss.

3. Results and discussions

3.1. Perforation test

Before and after the perforation test the FML specimens were

Fig. 4. Perforation test results for FML specimens prepared with 2024-T3 aluminum treated before (a and b) and after (c and d) the drilling test.

Fig. 5. Curves obtained for samples with (a) untreated aluminum, (b) treated, and (c) a comparison between both conditions.

photographed in order to compare the samples produced with treated and untreated 2024-T3 aluminum. Fig. 4 shows the samples before and after the perforation test.

The most used method to evaluate a material submitted to the perforation test is the force curve as a function of the displacement. Fig. 5 shows (a) the curves obtained for the samples with untreated aluminum, (b) the curves obtained with the treated aluminum and (c) a comparison between both conditions.

After analyzing Fig. 5 (a) and (b) it can be noticed that all samples presented similar failure mechanisms, in addition Fig. 5 (c) shows that the treated samples showed a slight decrease in the recorded maximum force. Thus, to better analyze this effect, all maximum forces were obtained, and an average was calculated. The untreated samples had a maximum force value (F_{max}) of 13.7 \pm 0.3 kN, while the treated samples had a value of 12.2 ± 0.4 kN. This behavior demonstrates a small decrease of approximately 10%, indicating that the treatment, by increasing adhesion at the metal/composite interface contributes to a decrease in the impact strength of the composite.

In addition, the displacement (I_p) was determined when the

maximum force decreases by half ($F_{max}/2$), for samples produced with untreated aluminum the value found was 7.3 ± 0.6 mm, while for the samples produced with anodized aluminum the value was 7.7 ± 0.2 mm. That is, there is a slight increase (5.5%) in the Ip value for anodized samples.

Finally, the area below the curves (up to I_p) was integrated to analyze the energy absorbed during impact. The calculated values for the untreated samples were 59.9 \pm 0.7 J and 57.6 \pm 0.3 J for the treated samples. Thus, there was practically no change in the absorbed energy values (\sim 4%) in both conditions.

The values found for both maximum strength and absorbed energy are slightly higher than those found by Hannemann et al. [[8](#page-10-0)], who performed the perforation test on hybrid carbon fiber/metal fibers/epoxy plates and found values maximum power and absorbed energy of 5 kN and 53 J, respectively. This behavior is due probably to the aluminum plates that make the composite much more rigid and tenacious at the same time.

In addition, Matthew Bondy and William Altenhof [\[9\]](#page-10-0) have been shown to have a maximum energy absorbed value of approximately 20 J

Fig. 6. Force curves as function of the time of each impact energy employed.

Fig. 7. Plates impacted with 40 J (a) with untreated aluminum and (b) with treated aluminum.

in the case of CF/polyamide composites. In addition, Yentl Swolfs et al. [[10\]](#page-10-0), who worked with CF and GF/epoxy hybrids achieved maximum absorbed energy values of only 13 J.

After comparing the values found in the present work and with the last articles highlighted, it is evident the advantage of adding metal to the constitution of the composites, the energy absorbed during the impact is more than the double. That is, an in-flight impact that would cause a complete failure of a composite part would only damage an FML and would not cause catastrophic failure of the structure.

3.2. Impact test

The samples produced with treated and untreated aluminum were impacted in different energies to compare the electrochemical treatment as a function of the extent of the damage caused to the samples. Fig. 6 shows the force curves as function of the time of each impact energy. As seen in the figure, it is important to note that each sample received only one impact.

Fig. 7 shows two images of the 40 J impacted plates, (a) the first one produced with untreated aluminum and (b) the second one produced with treated aluminum. It can be seen the cavities caused by the impact.

To evaluate the extent of the damage, two characterizations were performed, the optical profilometry and the optical microscopy of the cross section of the plate.

3.2.1. Optical profilometry

Optical profilometry was performed to evaluate the depth and volume of cavities caused by the impact test. [Fig. 8](#page-5-0) shows some analyzed cavities (a) 9 J, untreated; (b) 9 J, treated; (c) 40 J, untreated; (d) 40 J, treated.

The volumes of the cavities were measured and are shown in [Fig. 9](#page-6-0). The data in yellow represent the samples produced with untreated aluminum, while the data in blue represent the samples produced with anodized aluminum.

Analyzing [Fig. 9](#page-6-0) it is noticed that from 16 J the volume of the cavity of the samples produced with the anodized aluminum are slightly larger. For better visualization of this phenomenon [Table 1](#page-6-0) was produced, in it is presented the percentage comparison of the depth and volume of the cavities.

Analyzing [Table 1](#page-6-0), it can be concluded that for low impact energy values (less than 12 J), the electrochemical treatment of aluminum benefits the impact resistance of the final composite. However, above a certain value (16 J) the treatment slightly decreases this resistance. This is because, the samples produced with the anodized aluminum have a more cohesive interface, which generates compressive forces at the time of the cooling of the material. These factors contribute to promote greater damage to the impacted composite, as seen by Xin Li et al. [\[11](#page-10-0)], who evaluated the influence of adhesion on the Ti/CFRP FML interface and noted that composites with better adhesion showed lower impact strength and absorbed less energy during the test.

It is noteworthy that the impact test is in accordance with the perforation test (using energy of approximately 200 J), in which a lower impact strength was noted for the samples produced with the treated aluminum. Despite these lower results of impact resistance for the treated samples, it must be taken into account the increased interfacial adhesion of the aluminum to the CF/epoxy composite. This increase was investigated by shear analysis.

3.2.2. Optical microscopy

In addition to optical profilometry analyses, optical microscopy was conducted with the purpose of evaluating the extension of damage caused after the impact test. It should be noted here that for high impact energies (30 J and 40 J), the samples during sample cutting (see [Fig. 3\)](#page-2-0) delaminated completely. With this, it should be taken into account that

Fig. 8. Analyzed cavities by optical profilometry (a) 9 J, untreated aluminum; (b) 9 J, treated aluminum; (c) 40 J, untreated aluminum; (d) 40 J, treated aluminum.

Fig. 9. Volume of impacted cavities measured by optical profilometry.

the best way to measure the damage caused by the impact is optical profilometry, and the macroscopy was presented as an auxiliary characterization. [Fig. 10](#page-7-0) shows the cross sections of the samples impacted with 9 J–25 J.

Analyzing [Fig. 10](#page-7-0)(a–d) it is noticed that the treated samples (b and d) did not show delamination, whereas the untreated samples (a and c) showed small damages (indicated by the arrows). However, considering [Fig. 10\(](#page-7-0)e–h) this phenomenon is reversed, the samples produced with treated aluminum (f and h) showed much more significant damage than the samples produced with the untreated aluminum (e and g). Stands out the sample produced with treated aluminum and impacted with 25 J (h), the aluminum cracked.

This analysis is in line with the characterization of profilometry, in which a higher impact resistance of the samples produced with treated aluminum was observed for lower energies (less than 12 J), but a lower resistance was noticed to higher energies (above 16 J).

3.3. Compression shear test (CST)

Six composite specimens in each condition, produced with treated and untreated aluminum, were subjected to compression shear test to evaluate the influence of anodization on shear strength. The average compression shear strength of the samples produced with untreated aluminum was 10.2 ± 1.5 MPa, while the compression shear strength of the samples produced with anodized aluminum presented a value of 11.9 ± 1.6 MPa. That is, the electrochemical treatment increased 16.7% the compression shear strength of the FML. [Table 2](#page-8-0) presents the comparative values of shear strength by compression found in the literature.

Comparing the values obtained through the CST test with the values found in the literature, it is noticed that when compared to FMLs they are equivalent. However, compared with values of the composite of Polyamide/CF they are inferior [[6](#page-10-0)]. This is because, the metal/composite interface is a critical region where the cracks start and propagate preferentially.

3.3.1. Optical microscopy

After the compression shear test, the samples were embedded in Bakelite and sanded until 1200 mesh for evaluation in the stereoscope. The images obtained by such technique are presented in [Fig. 11.](#page-8-0)

Analyzing [Fig. 11](#page-8-0) (a) and (b) it is noticed that the samples produced with the untreated aluminum delaminated at the aluminum/composite interface, as highlighted by the white arrows. However, the samples produced with anodized aluminum, [Fig. 11 \(c\) and \(d\)](#page-8-0), presented the failure (white arrows) at the carbon fiber/epoxy interface, evidencing an unaltered aluminum/composite interface (red arrows).

3.4. Interlaminar shear test (ILSS)

Another mechanical test that evaluates interlaminar shear strength is ILSS. In this test, 12 samples were used for each condition. After calculating the interlaminar shear strength average, the samples produced with untreated aluminum showed a value of 57.4 ± 2.3 MPa, while the samples produced with anodized aluminum presented a value of 64.5 ± 1.2 MPa. Anodization increased interlaminar shear strength by 12.4%. [Table 3](#page-8-0) presents the comparative values of interlaminar shear strength found and some values present in literature.

Comparing the values obtained through the ILSS test with the values found in the literature ([Table 3](#page-8-0)), it can be observed that the FML composite produced with anodized aluminum is the one that presents greater resistance to interlaminar shear. Even if compared with a conventional composite without metal plates the FML produced in this work presents higher values [[14\]](#page-10-0). Indicating the quality of the anodizing process.

The values of the interlaminar shear strength found for the FML produced with anodized aluminum are in agreement with the normal resistance values of conventional CF/Epoxi composites [[18\]](#page-10-0), that is, the

Fig. 10. Cross sections of impacted FML samples: (a) 9 J, untreated; (b) 9 J, treated; (c) 12 J, untreated; (d) 12 J, treated; (e) 16 J, untreated; (f) 16 J, treated; (g) 25 J, untreated; (h) 25 J, treated. $20\times$ magnification.

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Table 2

Compression shear strength obtained by CST test and values found in literature.

metal/composite interface is no longer the most critical FML interface.

3.4.1. Optical microscopy

After the interlaminar shear test the samples were embedded in Bakelite and sanded until 1200 sanding sandpaper to evaluate their failure mode. The images obtained by such technique are present in Fig. 12.

Comparing Fig. 12 (a) and (b) it was not notice large differences between them. In both cases the aluminum/composite interfaces remained adhered, failures occurred at the carbon fiber/epoxy interface (white arrows). However, it is clear that the failure mode occurred by interlaminar shear, thus validating the values found by the ILSS technique.

3.5. Iosipescu test

The Iosipescu test was carried out with the purpose of evaluating the influence of the electrochemical treatment on the translaminar resistance of the metal fiber laminates produced. The test was not carried out until the total rupture of the samples, after the first force decrease perceived by the software, the load application was ceased. That is, after the first crack propagated and the resistance of the sample was reduced,

Table 3

Interlaminar shear strength obtained through the ILSS test and comparison with literature.

Material	Tensile Stress (MPa)	Reference
CF/PPS	58.4	[14]
$Ti_A/CF/Epoxi$	$36.4 + 2.4$	[12]
Ti/CF/Epoxi	$56.0 + 10.0$	151
GI.ARE@	$40.9 + 4.2$	F161
CARAI.J.R	$38.1 + 1.2$	[17]
Al/CD/Epoxi	$57.4 + 2.3$	
Treated Al/CF/Epoxi	$64.5 + 1.2$	

Fig. 11. Cross sections of samples tested by CST (a and b) untreated; (c and d) treated; 20 \times magnification.

Fig. 12. Cross sections of the samples tested by ILSS: (a) untreated and (b) treated. $20 \times$ magnification.

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Table 4

Iosipescu shear strength and comparison with values found in literature.

the test was stopped. Seven samples in each condition were tested.

The samples produced with untreated aluminum presented a shear strength average of 176.2 ± 10.8 MPa, while the samples produced with anodized aluminum presented an average of 192.5 \pm 2.8 MPa. The electrochemical treatment increased the translaminar shear resistance by 9.2%. Table 4 presents the comparative values of interlaminar shear strength found in literature.

As in the ILSS test, the values obtained by the Iosipescu test were higher than the values found in the literature (Table 4), it is observed that the FML produced with anodized aluminum is the one with the highest resistance to translaminar shear (192.5 MPa). In addition, the decrease of the standard deviation for the samples produced with the treated aluminum (2.8 MPa), when compared to the non-anodized samples (10.8 MPa), approximately five times lower, stands out. Corroborating the higher quality of the treated FMLs.

3.5.1. Optical macroscopy

After the Iosipescu shear test, the samples, with and without any previous treatment, were evaluated by stereoscopy. The images obtained from the treated and untreated samples are shown in Fig. 13.

Comparing Fig. 13 (a) and (b) with Figures (c) and (d) a large difference can be noticed between the composites produced with treated aluminum and produced with untreated aluminum. While the nonanodized aluminum totally delaminated at the metal/epoxy/CF interface, the composites produced with treated aluminum presented only minor failures.

It is important to observe that of the all samples produced with aluminum as received, only three did not show total delamination of at least one composite metal interface (samples 1, 2 and 4), all others were delaminated during the mechanical test. That is, the treatment makes possible the production of the Al/CF/Epoxi FML by hot compression molding.

4. Conclusions

After the accomplishment of the experiments, it was possible to reach the initial objective: to evaluate the influence of the electrochemical treatment (of the aluminum plates used in the composite manufacture) on the impact resistance and shear strength of Al/CF/Epoxy laminates.

The perforation test indicated a small decrease $(-10%)$ for samples produced with anodized aluminum, indicating that the treatment, by increasing the adhesion at the metal/composite interface contributes to decrease the impact strength of the FML. The impact test corroborated this observation for impact energies above 12 J. The most likely explanation for this is that the samples produced with the anodized aluminum have compressive forces at the aluminum/CF/Epoxy interface, generated during the processing of the material. Such forces contribute to a decrease in the impact resistance of the FML.

However, the characterization of the interface indicated an increase in adhesion in the interface in question. The CST indicated a 16.7% increase in compression shear strength, while ILSS indicated a 12.4% increase in shear strength resistance, finally Iosipescu indicated a 9.2% increase in translaminar shear strength.

Additionally, most of the plates produced with untreated aluminum delaminated after a few weeks in stock due to internal stresses from the production process associated with low adhesion at the Al/CF/Epoxy interface.

Thus, despite the decrease in the impact resistance, the Al/CF/Epoxy interface was superior after the electrochemical treatment with phosphoric acid. Since the treatment made possible to obtain the composite AA2024 T3/CF/Epoxy FML.

Fig. 13. Cross sections of the Iosipescu tested samples (a and b) untreated; (c and d) treated. 6.5 \times magnification.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Alberto L. Santos: Conceptualization, Methodology, Writing - review & editing. **Roberto Z. Nakazato:** Methodology. **Sebastian Schmeer:** Methodology. **Edson C. Botelho:** Supervision.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at [https://doi.](https://doi.org/10.1016/j.compositesb.2019.107718) [org/10.1016/j.compositesb.2019.107718.](https://doi.org/10.1016/j.compositesb.2019.107718)

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