ENVIRONMENTAL CONDITIONING EFFECTS ON THE MECHANICAL PROPERTIES OF TITANIUM FIBER-METAL LAMINATES

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1 Abstract

Fiber metal laminates (FML) have been particularly attractive for aircraft structures. While weight reduction and improved damage tolerance characteristics were the prime drivers to develop this new family of materials, it turns out that they have additional benefits which become more important for today's engineers, as the high mechanical strength and improved safety. Hence the combination of these aspects in one material is an extraordinary achievement. The specimens of FMLs were prepared with titanium plates and carbon fiber/epoxy resin prepreg. This study aims to evaluate the effect of two different environmental conditions (hygrothermal and thermal shock) on the mechanical and elastic properties of titanium FML, since this environmental effects should always be considered in the design of structural components, when these materials are submitted to the effects of the atmosphere and high mechanical efforts.

2 General Introduction

Since from the 1950s, when the modern transport aircrafts were introduced in the civil aviation, has been observed the in-service structural failures. So, it has focused the minds of materials engineers and designers on methods for improving flight safety. Furthermore, the high maintenance costs for airliners have generated a strong necessity for more durable and more damage tolerant structural materials, to reduce the maintenance costs and the structural weight of the aircraft. Fiber metal laminates (FML) were primarily developed for fatigue prone areas of modern civil aircraft. However, several grades of this material offer additional advantages such as damage tolerance, fire and impact resistances [1, 2].

This significant generation of composite materials consisting of stacking layers of polymeric composites alternately with metal plates, forming a sandwich structure (Fig. 1), where the outer layers are composed of sheet metal [2, 3].

![Fig. 1. Fiber metal laminates (FML) [3].](image-url)
In 1984 a patent on the aramid FML has been deposited in the United States and filed in 1993 by ALCOA, having as inventors of this new material: Schijve, and Vogelesang Marissen. However, the first commercial product was developed in mid-1982 under license from ALCOA. The production of this material occurred only in 1984, and had its official presentation in the market at the Paris Air Show 1985 [3].

ARALL® laminates have been developed from the use of aramid fibers embedded in an epoxy structural adhesive between multiple layers of sheets of aluminum alloy [4]. The first manufactured parts with ARALL® in the aerospace industry were panels wings Focker 50, where it was proven weight reduction of 20% compared to the original design aluminum [3]. The ARALL® has been available on the market in 1987 and in 1988 the French company Aerospatiale presented their studies of the application of ARALL® in the fuselage of the Airbus A320. However, the result has been catastrophic, because the ARALL® proved to be unsuitable for use in airframes, since aramid fibers present low compression resistance [3].

From these results, it has come the need to develop a new fiber-metal laminate for aeronautical applications. Thus was born the GLARE® developed in partnership with the University of Delft, the Netherlands. On October, 1987, a patent was filed by Akzo, and only in 1991, has been marketed by the union of Akzo (third) and Alcoa (2/3) [5,6,7]. Since its development, has been found in GLARE® not only material with excellent fatigue properties, but also other interesting properties of impact resistance and residual shear strength, fire resistance and corrosion resistance when compared to monolithic aluminum, due to sandwich structure of fiber-metal laminates, which is interspersed with plates of sheet metal and polymer composite [3].

The development of GLARE® was based mainly on the high load break that glass fibers could support, for applications in coatings fuselage aircraft, this being the largest differential of the GLARE® compared to ARALL® [3,8]. The first component in the plane that flew with GLARE® has been C5-A Galaxy by the USAF in 1995, already the first application in the civilian sector has been in the cargo hold of the Boeing B 777 and in the cavern pressure of Bombardier Learjet 125. In applying for fuselage panels, Airbus has lead this market and the design Airbus A380 is a good example, being the first aircraft to use the GLARE® in the primary structure of a surface, with approximately 400m², allowing a reduction in weight 25%, with savings of U.S. $280.00/kg reduced [3].

The laminate of the third generation of this new class of materials is called out CARALL®, which has been developed with carbon fibers, using the same type of processing to ARALL® and GLARE® laminates. The combination of high mechanical strength and stiffness with good impact properties provide great advantages to this laminate for aerospace applications [8]. However, the production of the CARALL® laminate has been limited, due mainly to problems associated with galvanic corrosion, from the contact between the carbon fiber and aluminum alloy [3].

Currently, ARALL®, GLARE® and CARALL® are being considered for a wide variety of aerospace applications. However, in future aerospace applications probably will require materials able to withstand higher temperatures for long periods of time. Thus, hybrid laminates for use in such applications should be developed based on materials that provide the ability to be used in higher temperatures [9].

The requirements for supersonic aircraft structural materials whose estimated speed cruising is 2.4 times the speed of sound (Mach 2.4), require materials that can withstand long term operation at temperatures close to 165°C, and further operations short term to 190°C. For a useful life of 25 years, it is estimated that the materials that make up the aircraft externally would be subjected to approximately 35,000 thermal cycles [10].

Some properties of titanium make of this hybrid titanium-carbon material a more attractive alternative for supersonic applications compared to GLARE® and CARALL®. The titanium has a coefficient of thermal expansion relatively low and the best relation between mechanical properties and weight [10]. These peculiarities make of titanium a good choice to act in conjunction with carbon fiber composite in supersonic applications [10].

The FML with titanium was originally designed for critical applications in fatigue, but this material was also able to offer high durability at temperatures above 350°F (177°C) and being suitable for applications in motors and supersonic structures [5]. The Boeing Company refers to this class of materials as TiGr (which stands for Titanium Graphite), and others has referred to these as HTCL (Hybrid Composite Laminates Titanium) [2, 11].

The first hybrid composite of fiber-metal with high temperature resistance has been developed by
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Langley Research Center - NASA, in order to meet requirements for high speed and high temperature (177°C) in flight. The initial test results were discussed in partnership with the Georgia Institute of Technology - Atlanta. This work was started in the 90s and was extremely promising. Then, extensive tests were initiated between Edward Li of the Boeing Company, W. S. Johnson of the Georgia Institute of Technology - Atlanta and NASA, in 1996, in order to evaluate the fatigue strength of laminate the titanium base at elevated temperatures. However, the test results presented significant amounts of delamination between the layers as a result of the development of fatigue crack in the titanium layer, or when a fatigue crack formed on the titanium evolved the damage rapidly in the interface between the layers of titanium/polymeric composite [2,12,13].

Thus, a major concern regarding the laminate titanium was the durability of the bond between the layers of polymeric composite and titanium plates [13].

Currently, hybrid titanium laminates are being studied in several countries, such as Netherlands, USA, Japan, Germany, Portugal, England and Brazil. In these works have been performed evaluating different heat treatments in titanium, different adhesives for bonding between the layers and different thermoplastic matrices (due to the possibility of its use to higher operating temperatures), seeking to improve the manufacturing process of these laminates, evaluating parameters of damage tolerance, as fatigue crack growth, delamination between layers, and studying the different influences of the environment on the mechanical properties of these laminates.

The fuselage of the aircraft are always exposed to environmental conditions such as atmospheric humidity and temperatures ranging from -69°F (-56°C) to 180°F (82°C), due mainly to long periods exposed to the hot sun, arid climates, polar climates and high altitudes. To ensure safety, materials engineers must be able to assess the influence of these conditions on the behavior of damage tolerance in aircraft structures. Without reliable predictive models to perform this type of analysis, materials engineers must rely on expensive experimental tests to demonstrate the safety of their materials and designs [14].

Therefore, it is extremely important to carry out experiments to assess the absorption of moisture from the atmosphere, which occurs both in the structure of the polymeric composite, in the polymeric composite/metal interface affecting directly the mechanical properties. In the fiber-metal laminate is believed that the effects of moisture can be minimized with respect to conventional composites, because in harsh environments the titanium protects the core of the polymer matrix composite of environmental effects such as moisture penetration and oxidation [13].

The thermal shock experiment evaluates the physical and mechanical properties after temperature variations flight envelope. In numerous cycles of thermal shock can occur thermal fatigue of the components of the material. This process of thermal shock can generate internal stresses that cause a variety damage forms, such as fracture in the polymer matrix, between the fiber and the matrix and delamination between the layers of the hybrid composite [8,15].

With the systematic increase in the structural use of composite materials, it is crucial to understand the mechanisms of damage and how they behave in the structure of the material. In this case, differences in the properties of materials under hygrothermal conditioning and under sudden changes in temperature are identified and assessed with the results of mechanical tests and viscoelastic behavior [15].

3 Materials and Methods

3.1 Materials

The fiber metal laminates of titanium (titanium FML) used in this study were processed and supplied by manufacturer Brazilian aircraft - EMBRAER S.A. The titanium sheets used for manufacturing this laminate has 0.40 mm thick and its composition is pure titanium (grade 4 according to ASTM B 265). In order to processing this FML material, the titanium sheets have been submitted to a superficial cleaning treatment for removing any impurities on Ti surface. These plates were stacked alternately with fabric carbon fiber/epoxy prepreg layers of approximately 0.38 mm thick, with the fibers oriented at 0/90° and a volume fraction of 55% fiber.

The designation used for the prepreg is F155/W3B282, supplied by HEXCEL Corporation. The laminates were manufactured by the hand layup process, where the titanium plates were arranged to form three layers of metal intercalated with two layers of polymeric composite.
The complete cure cycle has been programmed to operate at 121°C with 78kPa pressure and vacuum of 0.083MPa, with heating and cooling rates of 2.5°C/min. The cure cycle was obtained from previous work [10,16]. Figure 2 shows a graphic of the cure cycle used in the processing of the titanium laminate studied.

3.2 Hygrothermal Conditioning

The FMLs were subjected to hygrothermal conditioning in a climatic chamber in order to evaluate the influence of moisture combined with the cyclic variation of temperature (thermal shock). This hygrothermal conditioning was based on ASTM D 5229 M-04 for composites undergoing mechanical tests in humid conditions.

The samples were exposed to 176°F (80°C) and relative humidity of 90%. The average period of conditioning was of eight weeks, long enough for the material reach the saturation with moisture. After this conditioning, these specimens were conditioned in thermal shock chamber.

3.3 Thermal Shock Conditioning

Thermal cycling weathering was carried out on an Envirotronics two zone vertical thermal shock test chamber, Model TSV 5-2-2-2-AC. In order to simulate a flight envelop of an airplane, the composite was exposed to several cycles of 20 minutes at -50°C followed by 20 minutes at +80°C. The thermal cycling was performed for 1000 cycles.

By using this conditioning, two coupons were conditioned: dry titanium FML and titanium FML after hygrothermal conditioning.

3.5 Free Vibration Test

Elastic modulus of FMLs after the environmental conditionings has been determined by free vibration tests.

The measurement principle of this test consisted of recording the free vibrations of a prismatic cantilever excited by a pulse using a suitable hammer. With the assistance of special software data acquisition has been possible to measure the decay of the amplitude as a function of time as well as the vibrational modes of the material.

The acquired data were saved by an appropriate program. The test parameters were as follows: frequency analysis range 1000Hz; acquisition time of 200ms and frequency resolution of 5Hz. The amplitude decay was measured using an accelerometer 0.6g. The dimensions of the specimens and its mass were also measured. With this procedure we obtained two types of lines: one free vibration damping and a function of the frequency response of the material (FRF).

A theoretical analysis of internal damping and dynamic stiffness for aligned continuous fiber composite was developed based on micromechanics models for the complex moduli. The free vibration method results generally present a logarithmic damping (Δ) given by the equation 1 [10].

\[
\Delta = \frac{1}{n} \ln \left( \frac{\delta_1}{\delta_n} \right) \quad (1)
\]

where: \( n \) is the number of peaks; \( \delta_1 \) is the amplitude of the first peak and \( \delta_n \) is the amplitude of the final peak analyzed.

The storage modulus (\( E' \)) can be obtained according to equation 2 [10].

\[
E' = \frac{4\pi^2 f^2}{3I} \cdot \left[ M + \frac{33}{140} m \right] L^3 \cdot \left[ 1 + \frac{\Delta^2}{4\pi^2} \right] \quad (2)
\]

where: \( E' \) = elastic modulus; \( f \) = natural frequency; \( I \) = inertial moment; \( M \) = accelerometer weight; \( m \) = specimen weight and \( L \) = specimen length.

Assuming a linear system of degree of freedom 1, the frequency response function is a decaying of natural frequencies of the sample, which correspond to a typical "fingerprint" of vibration modes. The number of peaks in frequency of vibration (vibration
modes) and the shape of the response function are a direct result of the stiffness of the material. Thus, one can evaluate the damping of the laminate, ie the distance between the purely elastic behavior and viscous behavior pure.

3.4 Tensile Test

The specimens were performed in accordance with ASTM D-3039 standard, with the objective of determining the tensile strength values after the environmental conditionings.

The dimensions of the samples used were 200mm x 25.4mm.

4 Results

4.1 Hygrothermal Conditioning

The effects of moisture present in the atmosphere should always be considered in the design of structural laminates. Therefore, moisture can penetrate into the polymer matrix by means of the diffusion process until an equilibrium concentration is reached. According to the works available in the literature [17], the moisture absorbed into polymer matrix composites can reduce the strength and stiffness of the laminate due to the plasticizing effect of the matrix, with the weakening of the fiber/matrix interface. These decrease on the mechanical properties are particularly significant at elevated temperatures. Thus, the presence of moisture into composite can generate significant changes in the physicochemical characteristics of the matrix. Figure 3 shows the results from moisture absorption in the titanium FML until 60 days of exposure in a hygrothermal conditioning chamber.

By analyzing the curves of moisture absorption from this laminate can be seen that after approximately 60 days of conditioning, the material absorbs on moisture average of 0.12%. Also can be observed that after a certain period of exposure it is not observed an significant increase of moisture absorption, indicating that the equilibrium was reached. This stabilization occurred after about 1200 hours or 50 days of exposure to hygrothermal conditioning.

The moisture absorption in this laminate is lower than that one observed in neat epoxy resin or in carbon fiber/epoxy laminates since the literature report values in these cases from 1.0 to 3.5wt% [18]. This evidence proves the performance of the outer layers of titanium, acting as barriers to the diffusion of moisture in the laminate.

4.2 Tensile Tests

The tensile tests are the main form of assessment of mechanical properties of structural composites in short-term trials and static requests. From the results obtained in the tensile tests recorded by means of stress-strain curves can be obtained variables such as tensile modulus, tensile stress and strain at yield point and ultimate stress [10].

In this work it was analyzed the tensile properties of the titanium FML without conditioning and after to be submitted to thermal shock and hygrothermal conditionings. Table 1 presents the results of the average values of tensile strength, tensile modulus and deformation of titanium FML, comparing this properties with these one observed by Glare®, Carall® and their constituents, as obtained in our previous works [19-20].

Figure 3. Moisture absorption results.
By using the results from Table 1, it was observed that all samples showed a similar behavior and it was noticed brittle fracture, with catastrophic fracture characteristics, in all analyzed cases. This behavior is due mainly to the contribution of ceramic fiber reinforcements. After this event, the curves show clearly that just happens to be requested titanium during the test.

From these results can be observed that the tensile strength of the pure titanium is less than the titanium FML (~74%). Also it was observed that tensile strength of titanium FML is higher than the values observed for GLARE® and CARALL® (~195% and 76%, respectively). Concerning about the modulus of elasticity, titanium FML also present higher value when compared with the another FML laminates, due to the titanium sheets contribution.

Table 2 shows the tensile properties values from titanium FML after to be submitted to environmental conditionings. As can be observed, both the tensile strength and the elastic modulus did not change whereas the standard deviation when compared unconditioned and conditioned specimens.

This small variation for ultimate tensile stress and modulus of elasticity may be in function of some defects generated during the environmental conditioning, degrading the interface and causing probably few fiber/matrix and composite/metal delamination. Figure 4 presents the titanium FML specimens after tensile tests.

![Titanium FML specimens after tensile tests](image)

**Table 1. Comparison between the tensile results of GLARE, CARALL, titanium FML, Ti and carbon fiber/epoxy laminate.**

<table>
<thead>
<tr>
<th>MATERIALS</th>
<th>TENSILE STRESS (MPa)</th>
<th>TENSILE MODULUS (GPa)</th>
<th>TENSILE STRAIN (%)</th>
<th>REFERENCE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon fiber/epoxy</td>
<td>1160 ±37</td>
<td>67.2 ±4.0</td>
<td>1.7 ±0.1</td>
<td>[19]</td>
</tr>
<tr>
<td>Titanium</td>
<td>550</td>
<td>106.80</td>
<td>15</td>
<td>[20]</td>
</tr>
<tr>
<td>GLARE®</td>
<td>380 ±23</td>
<td>55.3 ±2.0</td>
<td>1.9 ±0.1</td>
<td>[19]</td>
</tr>
<tr>
<td>CARALL®</td>
<td>420 ±29</td>
<td>58.9 ±2.0</td>
<td>1.6 ±0.2</td>
<td>[19]</td>
</tr>
<tr>
<td>Titanium FML</td>
<td>742 ±38</td>
<td>89.3 ± 3.0</td>
<td>1.5 ± 0.3</td>
<td>[16]</td>
</tr>
</tbody>
</table>

**Table 2. Tensile results of titanium FML.**

<table>
<thead>
<tr>
<th>TITANIUM BASED FMLs</th>
<th>TENSILE STRESS (MPa)</th>
<th>TENSILE MODULUS (GPa)</th>
<th>TENSILE STRAIN (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unconditioned</td>
<td>742.00 ± 38.00</td>
<td>89.30 ± 3.00</td>
<td>1.5 ±0.3</td>
</tr>
<tr>
<td>Thermal shock</td>
<td>620.09 ± 11.90</td>
<td>80.71 ± 11.67</td>
<td>2.5 ±0.2</td>
</tr>
<tr>
<td>Hygrothermal/thermal shock</td>
<td>584.71 ± 20.29</td>
<td>81.58 ± 3.19</td>
<td>2.5 ±0.2</td>
</tr>
</tbody>
</table>

**Fig. 4. Tensile stress failure behavior in titanium FML.**
4.3 Free Vibration Tests

Figure 5 shows a representative result obtained from free vibration test (profiles of the curves of frequency response function titanium FML) without conditioning. The behavior of these curves was similar for the conditioned specimens.

From Figure 5a is obtained the first vibration mode (natural frequency). From Figure 5b can be obtained the amplitudes and the oscillation numbers between these peaks. By using these data and the equations 1 and 2, can be calculated the storage ($E'$) and the loss ($E''$) moduli. These values and the tan δ value are shown in Table 3.

From Table 3 can be noted the occurrence of a decrease in vibration damping compared to the laminate without environmental conditioning, mainly when this is subjected to thermal shock conditioning. This result may be confirmed by a decrease in the tan δ value (loss factor), which corresponds to the internal damping and friction of the material. Note that the decrease in the value of tan δ in the samples that were subjected only to cyclical variation temperature was approximately 28%, due to a decrease of loss modulus ($E''$).

It is also observed a decrease in the storage modulus ($E'$) values, around 43% in both environmental conditionings, indicating that these conditionings may have caused variations in the stiffness of the hybrid laminates. This behavior can be explained by a possible reduction in energy loss factor of the laminate (tan δ and $E''$) and a reduction in storage modulus ($E'$), causing a higher exponential decay between the maximum amplitudes of the vibration peaks associated with an increased frequency of vibration.

The increase in delaminations in the samples according to the environmental conditioning would increase friction between the interfaces, and it would increase dissipation energy, however, this behavior was not observed.

The reduction of moduli for the samples subjected to hygrothermal conditionings followed by thermal shock may be explained due to the possible plasticization of the matrix caused by the absorption of water in the conditioning, causing a swelling of the resin, thus increasing the moving among the polymeric chains. Also should be considered that the interface between titanium and carbon fiber/epoxy prepreg usually is poor, contributing to this behavior.

Figure 5. Representative damping behavior curves obtained from titanium FML.
5 Conclusions

The influence of thermal shock and hygrothermal conditionings on mechanical and viscoelastic properties of titanium/carbon fiber/epoxy laminates have been investigated. Results obtained by tensile tests showed that this laminate presents tensile stress and tensile modulus higher than Glare® and Carall® laminates.

From the hygrothermal point of view, it was observed a lower moisture absorption due to the presence of Ti sheets, but when associated with thermal shock conditioning, the mechanical and viscoelastic properties decreased, indicating that this combination is more damaged between the conditions studied in this work. Therefore, when compared the worst situation (hygrothermal and thermal shock associated) of titanium FML with Glare® and Carall®, the mechanical properties values are still higher indicating that this FML is a good candidate in order to replace aluminum for aerospace application.

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