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CFRP STRUCTURAL CAPACITORS: EFFECT OF DAMAGE AND MECHANICAL LOAD ON CAPACITANCE

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Sommario

Scopo del presente lavoro è di studiare l'effetto delle sollecitazioni meccaniche e dell'eventuale danneggiamento sulle performance di capacitori strutturali ottenuti da laminati compositi CFRP, mediante introduzione nel piano medio di uno strato di dielettrico PET trattato con soda caustica. Tali capacitori sono stati dapprima caratterizzati meccanicamente mediante prove di trazione e prove di delaminazione, e i risultati comparati con quelli del laminato CFRP privo di dielettrico. Mediante misura della capacità elettrica prima dell'applicazione del carico meccanico, sotto carico e dopo la rimozione del carico è stato mostrato che la capacità diminuisce per alti livelli di deformazione, a causa del danneggiamento progressivo delle lamine di CFRP evidenziato anche attraverso analisi frattografiche appositamente eseguite. Il fenomeno è pero reversibile in quanto dopo la rimozione del carico la capacità torna al valore iniziale. L'analisi sperimentale ha mostrato che le performance meccaniche di tali capacitori strutturali sono limitate da cedimenti prematuri legati alle tensioni interlaminari che si hanno all'interfaccia tra CFRP e dielettrico. Sono state eseguite pertanto simulazioni numeriche al fine di valutare lo stato tensionale ai bordi liberi e definire così un criterio che possa essere utilizzato in fase di progettazione per una accurata previsione della resistenza di tali materiali multifunzionali.

Abstract

Aim of this work is to study the effect of mechanical load and damages, on the performance of structural capacitors, made by CFRP composite laminates with a PET dielectric film (treated with sodium hydroxide) inserted at the laminate middle-plane. Such capacitors have been characterized by ILSS and tensile tests and the properties so estimated were compared to those of the simple CFRP. By measuring the capacitance before mechanical loading, under loading and after unloading, it has been observed that, due to the damage of the CFRP layers, proved also by proper fractographic analysis, at high strain level the capacitance decreases although it exhibits a complete recovery after unloading. Successive FEM analysis have been performed on structural capacitors to detect the interlaminar stress field and to implement a suitable criterion that can be used at the design stage to a reliable prediction of the failure load of such multifunctional CFRP composite materials.

Parole chiave: Structural capacitor; smart material; layered structures; free-edge stresses.

1. INTRODUCTION

In the last years the increasing use and study of fibre reinforced composite materials have resulted in very competitive lightweight structures, first in the aeronautical and aerospace field, then in automotive and marine applications. Reducing structural weight is one of the major ways to improve performance of any transportation system. Lighter and stronger materials allow greater range and speed and also contribute to reducing operational costs.Traditional materials used by the automotive industry cannot fulfill the lightweight requirements of future vehicles. Hence, to realize real alternatives to the combustion engine, two goals have to be reached:

- lightweight structures, in order to decrease energy consumption
- high electrical storage capability for increased range.

In order to keep up with the growing request of electrical vehicles, a different approach towards composite materials is desired, focusing attention on lightweight structures and novel multifunctional materials. Composite materials and multifunctional materials could reduce the structure weight and increase the energy stored allowing the batteries to vanish inside the structure.

In the laboratories of Imperial College London, several samples of multifunctional materials such as structural batteries and supercapacitors, have been demonstrated [1]. Two parameters are fundamental for energy storage: the energy density and the power density [2]. The first one defines the amount of energy that can be stored in a given volume or weight. The latter one defines the way this energy can be stored in the device. Batteries provide a high energy density with low power density, while capacitors offer a limited energy density with high power density. Supercapacitors are a compromise between batteries and capacitors.

At the end of the nineties the first idea of multifunctional thin structural capacitor materials was introduced by Luo and Chung [3], using two unidirectional CFRP layers as conducting plates and paper or polymer (which separate the two CFRP layers) as dielectric. By this design, the material is able both to carry mechanical load and to store electric energy, allowing the electronics to be introduced into the structure.

Recently, in the laboratories of Swerea SICOMP and in collaboration with Volvo Cars, several structural capacitors were built. The structural capacitors were made in the spirit of Luo and Chung, stacking four CFRP layers separated by different types of dielectrics such as polyester (PET), polyamide (PA), polycarbonate (PC) or paper [4]. Mechanical and electrical tests were performed to assess the multifunctional efficiency of the structural capacitors. In the mechanical characterization two types of mechanical test were performed. Tensile tests were performed to characterize the tensile properties and the three-point bending tests were performed to measure the interlaminar shear strength. It is widely known from literature that in composite laminates significant damaging (matrix micro craking etc.) starts typically when the applied overcomes 30-40% of the failure load. It can cause delamination between CFRP layers and the dielectric film. For composites laminates made by woven fabric Gao et al. [5] have demonstrated that the matrix cracks on the transverse layers often result in significant delamination in the region of the crimp, reducing also the stiffness of the composite.

The aim of the present study is to detect the influence of cracks and delamination on the electrical performance of structural capacitors made from CFRP composite laminates.

In detail, tensile test will be performed on electrically charged specimens to assess the behavior of the capacitance as function of the strain and the damage state. For this purpose a new setup and new type of specimens have to be developed. Such test shall be designed so that electrically charged specimens are isolated from the machine. Also, the specimen design will allow to avoid short-circuit between two adjacent carbon layers. Then fractographic analysis will be used to investigate damage mechanism in the specimens.

Furthermore, in order to implement a design method to predict the mechanical strength of similar capacitors, finite element simulations of the specimens tested will be performed to investigate the stress field at the free-edges and at the interface between the PET film and the CFRP layers.

2. MATERIALS AND MANUFACTURING

2.1 Material properties

The main part of the experimental analysis carried out in this study, has been performed in the laboratories of Swerea SICOMP. Fig. 1 shows a schematic representation of the structural capacitor considered:

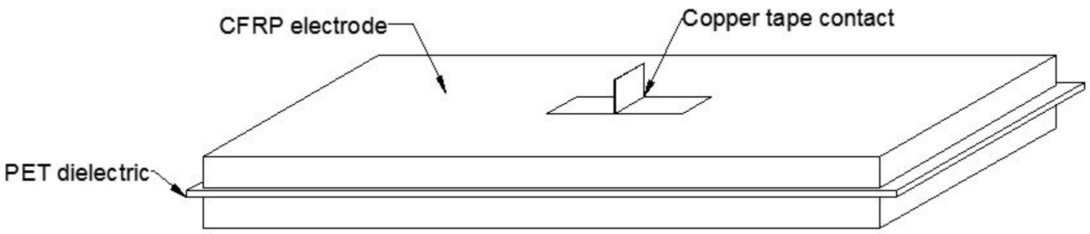

Figure 1: CFRP structural capacitor scheme.

In practice, the current work considers structural capacitor made by CFRP laminate with a PET film dielectric having a thickness of 50 µm. Considering all the structural capacitor materials tested in [4], the PET with 50 µm thickness was found to exhibit the highest capacitance, equal to 447 nFm^{-2}.

All the studies reported in literature, have been focused toward the optimization of the electric behavior and no one has yet evaluated the influence of mechanical loads and damage on the capacity of the structural capacitors.

Therefore, the research presented in this paper was performed with the objectives to develop a useful test method to characterize the effect of the mechanical load and of the damage on the capacitance of structural capacitors; the developed method has been applied to a new type of structural capacitors with dielectric made by a PET-film treated with NaOH.

In more detail, the electrodes layers were made from 0.125 mm thick prepreg woven. The prepreg was supplied by Umeco Structural Materials, UK. It was a 245 gm^{-2} 2 x 2 Twill HS [0°/90°] configuration, labelled MTM57/CF3202.

Carbon fibre layers are well suited for building capacitor plates. In fact, carbon fibres have high electric conductivity, mostly in the fibre direction whereas the epoxy matrix surrounding them is an insulator. Consequently, the electric conductivity in the transverse direction is much lower than in the fibre direction. Also, typically the electric conductivity in the out-of-plane direction is usually lower than the in-plane one [6].

As dielectric, a polyester film supplied by Trafomo AB(Sweden) was employed. It was, in practice, a 50 µm thick thermoplastic polyester film of DuPont Mylar A. Mechanical and electrical properties of such a 50 µm PET film and the prepreg based composite are provided in the following Table 1.

Table 1: Proprieties of prepreg MTM57/CF3202 and PET 50 µm film Mylar@ A

Material	E_L	E_T	σ_L^{ult}	σ_T^{ult}	ν_{12}	ν_{21}	ILSS	Dielectric strength
	[GPa]	[GPa]	[MPa]	[MPa]			[MPa]	[kV]
MTM57/CF3202 fabric	64.2	65.1	642	665	0.05	0.05	71.5	-
PET 50 µm Mylar@ A	3.8	3.8	190	190	0.44	-	-	7.1

2.2 Manufacture of structural capacitors

By using the above mentioned prepreg and PET film, two different laminates were manufactured: one for tensile tests and one for ILSS tests. The laminate for tensile tests was obtained by stacking two plies of prepreg on each side of a PET film, whereas the one for interlaminar shear strength (ILSS) test was made by eight plies on each side of the PET film. To achieve good mechanical and electrical properties, laminates were manufactured by using a vacuum bag system. Vacuum was applied to achieve void free and high quality laminates, since the presence of voids cause decrease of mechanical

properties, in particular ILSS (shear strength is very sensitive to voids). Also, electrical properties are likely to drop in presence of voids. Laminates were stacked in a coated mould with peel plies on both sides. This assures equal properties on both surfaces. The mould was sealed with vacuum bag using butyl tape. The general schematic illustration of the manufacture set-up is presented in Fig. 2.
Vacuum was applied at room temperature and 1 atm for 30 minutes. To complete the laminate's curing process, the mould was placed in an oven and heated according to the supplier's instructions (120 °C for 30 minutes).

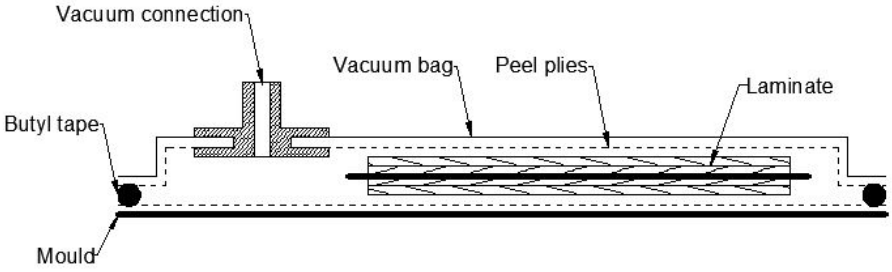

Figure 2: Vacuum bag set-up.

3. EXPERIMENTAL CHARACTERISATION

3.1 ILSS test

In order to evaluate the effects of dielectric film (inserted at mid-thickness) on the interlaminar shear strength of the laminates, ILSS tests were performed. As it is well known, ILSS test is a typical procedure for monitoring laminates quality and are suitable for the comparison of materials. Previous studies demonstrated noticeably negative effects of both non-treated and plasma treated PET films, on the interlaminar shear strength of the CFRP laminate. All the capacitors examined have shown ILSS significantly lower than that of the CFRP reference material.
The short beam three-point bend test (fig.3) was performed according to the ASTM D2344/D2344M standard [7], by using a MTS 20/M test machine with a 100 kN load cell. A crosshead speed of 1 mm/min was used. According to the standard, span-to-span measured thickness ratio is 4.0 with an accuracy of ±0.3 mm. Six specimens having a thickness of 5.85 mm, length of 36 mm and width of 12 mm, were cut from an a plate of 70 x 150 mm manufactured by the above described hand lay-up.

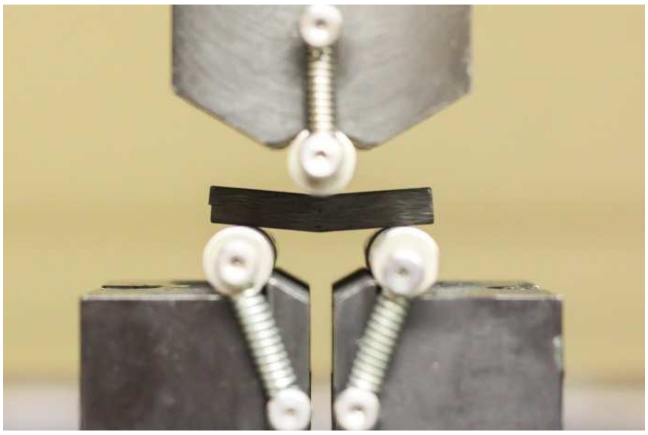

Figure 3: ILSS test set-up.

3.2 Tensile test

Tensile tests were performed at room temperature using an INSTRON 8501/H0162 with 100 kN load cell. A constant crosshead speed of 1 mm/min was used and the axial strain was measured using a 25 mm gauge length extensometer.

Tests were performed on two kinds of specimen: one made by only four layers of CFRP (thickness of 1.21 mm) and one made by four layers of CFRP and the PET dielectric at mid-thickness (capacitor, thickness of 1.27 mm).

3.2.1 Tensile test set-up for electro-mechanical test

In the start-up study different trial specimens were made from the same structural capacitor plate. These trials resulted in a procedure being developed for edge preparation. In particular, edges were polished with P800 and P1200 paper creating a small angle of approximately 30-45°, that should minimize the risk to short circuit after cut edges. Details of the polished edge are depicted in the following Fig. 4.

Figure 4: Edge detail of the specimens used for tensile test (transverse section).

By this edge preparation it is possible to achieve a capacitance equal to that of the plate, i.e 427.16 nFm^{-2}. An open circuit analysis (OCP) showed a low decrease of the capacitance with time; in detail the treated specimens exhibit a linear behaviour with a slope lower than that of untreated specimen, as it is possible to observe in Fig. 5.

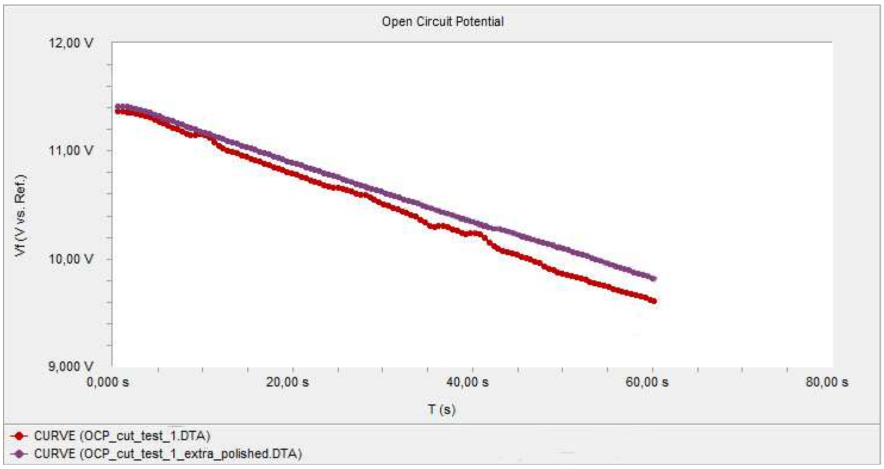

Figure 5: Open circuit test.

3.2.2 Tensile test under electrical charge and electrical properties

In order to obtain a better grip and to guarantee a perfect electrical insulation from the test machine, GFRP tabs were bonded to the specimens by using an epoxy adhesive (*Araldite 2012*, supplied by Huntsman®).

To characterize the electrical properties of the structural capacitor material, four different test were carried out:

- Test on original laminate;
- Test on specimens before loading;
- Test on specimens under mechanical load at a strain level between 0.6-0.65%;
- Test on specimens after unloading.

In detail, the first test was performed to characterize the entire laminate before cutting into specimens. Before mechanical loading, capacitance of each specimen was measured and then again under mechanical load. The under-load tests were performed in the strain range 0.6%÷0.65%. This is a reasonable level of strain because cracks appear at much lower strain.

After unloading and removing each specimen from test machine, capacitance was measured again to investigate residual effect of stress and damage on capacitance. All the electrical tests were performed using a *General Electric Program IDA200* with *keithley* 8009 electrode fixture. The capacitors were charged up to 10 V and then discharged by measuring the charge, in coulombs (C), from which the capacitance has been calculated.

3.2.3 Fractographic analysis

The fractographic analysis has been performed by using a digital microscope to detect the presence of different damage mechanisms, as cracks and delamination, as well as the cracks density for specimens tested under static tensile condition. Fig. 6 shows the set-up and the microscope used.

Figure 6: Analysis of damage mechanism under tensile load by using Dino-Lite Pro microscope.

After electrical/tensile test, specimens were removed from test machine and the edges were studied. Prior the fractographic examination the inclined edges were removed by polishing. Since specimens were not tested to failure, matrix cracks were not well visible in unloaded coupons. For this reason the specimens were loaded mechanically once again to identify damage.

Gao et al. demonstrated that transverse matrix cracks initiated through-out the bulk of the coupons [5]. Sectioning coupons showed that crack densities measured at the coupon edge did not differ from crack densities measured away from the coupon edge. In current study, cracks were identified and counted on the edge of the specimen loaded to a strain level of 0.6-0.65%, to obtain the crack density.

3.2.4 Out-of-plane tensile test

Due to the weak adhesion between the PET film and the CFRP layers, the out-of-plane tensile strength of the analyzed capacitors is quite low, compared with the corresponding performance of the simple CFRP. In order to determine the so called peeling strength (σ_z^{TF}) of the such capacitors, a proper out-of-plane tensile test has been performed. Set-up is designed aiming to obtain a uniform stress field in the out-of-plane direction. A specimen of 20x20 mm has been cut from the original plate and attached to proper aluminium tabs as it is shown in the following Fig. 7.

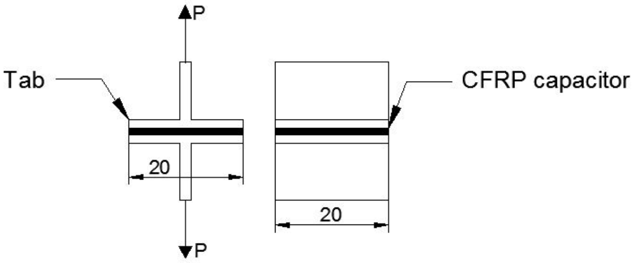

Figure 7: Out-of-plane tensile test specimen set-up.

4. RESULTS AND DISCUSSION

4.1 ILSS test

Results of ILSS tests were compared with those obtained by Carlson and Asp [4], who performed ILSS tests on similar structural capacitors obtained by using a 50 µm PET film treated with plasma and without treatment. Such a comparison shows that the performance of specimens with 50 µm thick film treated in NaOH, were slightly higher than those of film treated with plasma, see Table 2. An increase of 2.3 MPa (6.8%) was measured compared to specimens treated with plasma whereas an increase of about 5.0 MPa (14.1%) was obtained, compared to dielectric with no treatment.

Table 2: ILSS test results.

Dielectric film	**ILSS** [MPa]	**Gain** [%]
PET 50µmNaOH T.	34.33 ± 0.55	Ref.
PET 50µm Plasma T.	32.0 ± 1.1	7%
PET 50µm No treatment	29.5 ± 1.3	14%
CFRP Reference	54.4 ± 1.5	-37%

Also, from Table 2 it is seen how the ILSS of the CFRP reference was 20 MPa higher (37%), therefore the extra step in manufacturing caused by the soda treatment is not justified for ILSS. Furthermore, treatment with NaOH can be dangerous because, according with the instructions, concentration is quite high.

The curves reported in Fig.8 show with more clearness the results reported in Table 2. Shortly, all the CFRP capacitors exhibit a ILSS significantly lower than that of the CFRP reference so that it is possible to state that the such CFRP capacitors can be subjected to easy delamination phenomena under various loading conditions.

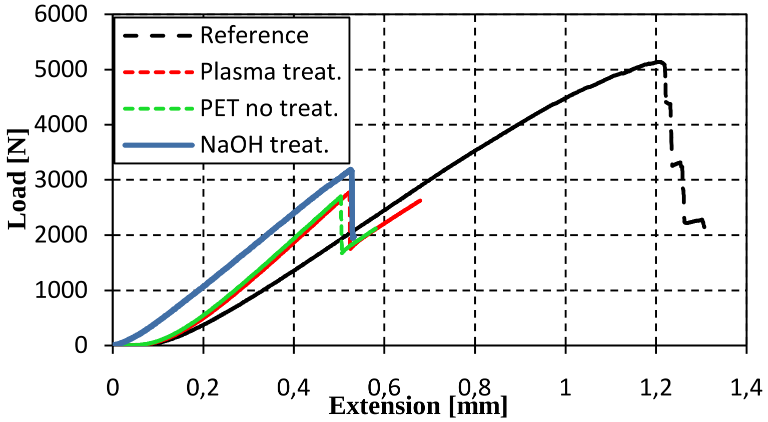

Fig. 8: Comparison of the results obtained from the various ILSS tests.

All the ILLS specimens analyzed have shown a typical shear/delamination failure at the CFRP-PET interface, as it is depicted in Fig.9. The red line indicates such a damage mechanism, whereas the blue line indicates the successive final bending failure.

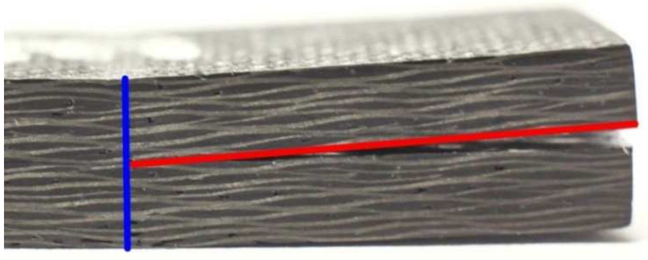

Figure 9: Typical failure of the ILLS specimens analyzed.

4.2 Tensile test

Results from the tensile tests are reported in Table 3 and compared with other surface treatments.

Table 3: Tensile tests results and comparison with independent results reported in literature.

Dielectric	**E** [GPa]	**σ_{ult}** [MPa]	**Gain** [%]
Pet- film 50 µm NaOH t.	49.2±1.80	501±25	-20%
Pet- film 50 µm Plasma t.	42.5±2.1	320±47	-49%
Pet- film 50 µm	42.7±3.0	354±66	-44%
CFRP reference	48.9±0.5	630±18	Ref.

From table 3 it is possible to observe how the soda treatment lead to a significant increase of the ultimate tensile strength. In particular, the ultimate tensile strength is now 56% higher than that of the laminate with the dielectric film without surface treatment, although is still 20% lower than CFRP reference.

The stress-strain curves reported in Fig.10 show that the strain to failure was in the range 0.95% - 1.10%, which is much higher than the 0.2% value measured for structural capacitors with plasma treated PET film [4].

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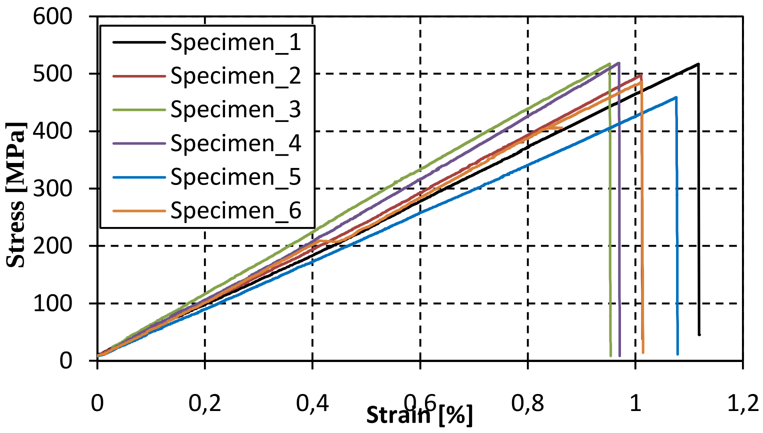


Figure 10: Stress-strain curves for NaOH treated laminates.

4.3 Capacitance under load

The experimental setup used for the measuring of the capacitance under load of the analyzed capacitors, is shown in the following Fig. 11.

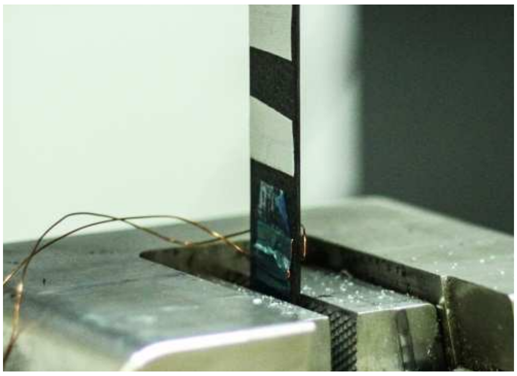
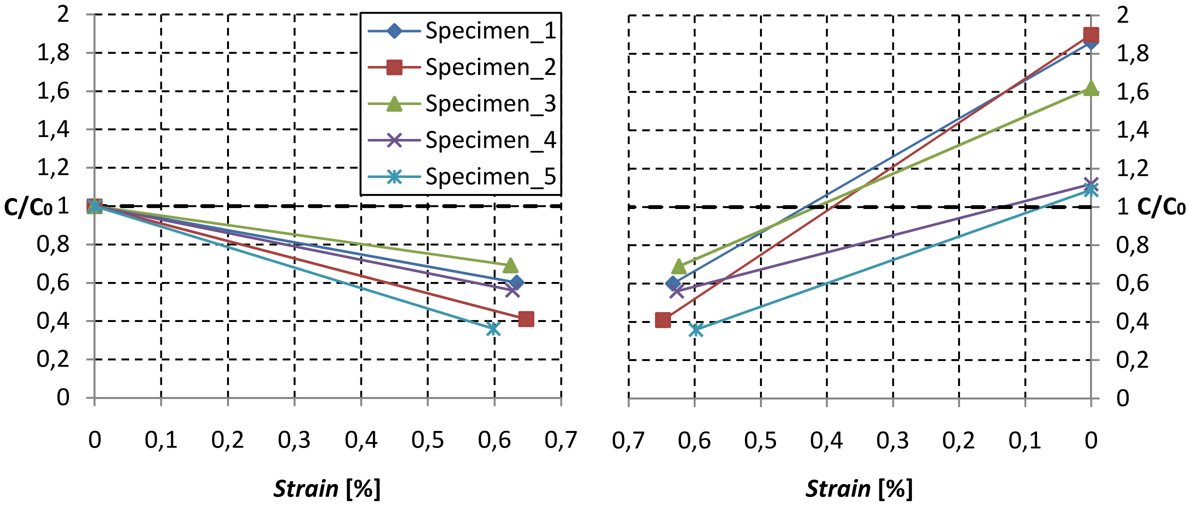


Figure 11: Experimental setup for capacitance measuring under load.

The application of the mechanical load leads to a drop of capacitance of about 50% (see fig.12), mainly due to the damaging of the CFRP layers. However, after unloading there is a complete recovery of the capacitance that, in some cases can reach also values superior to those of the virgin material (see fig.12). Such a particular behaviour is not understood at this stage and must be studied further.

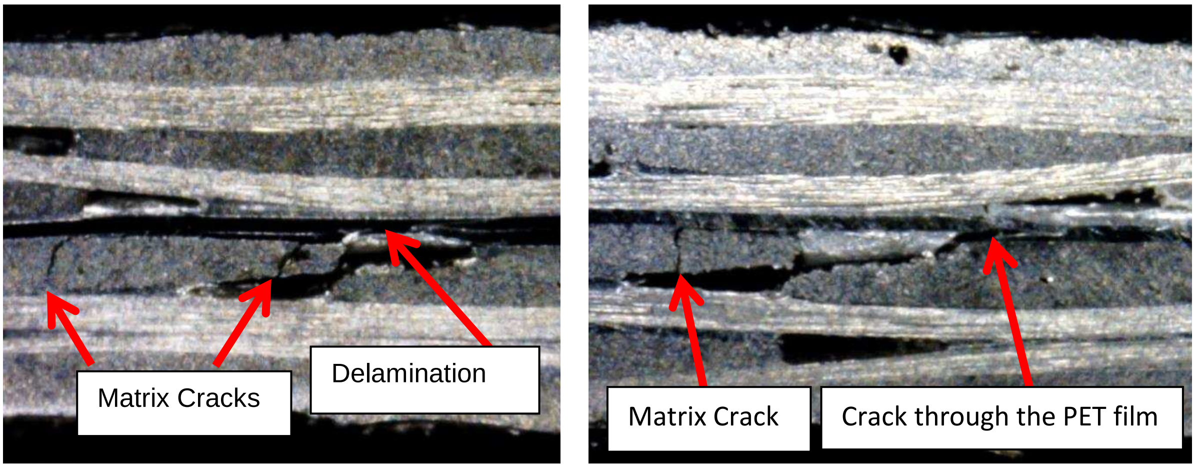


(a) (b)

Figure 12: Capacitance-strain curves during loading (a) and unloading (b).

4.4 Fractographic analysis

The fractographic analysis performed has shown that matrix cracks develop along the weft tows, parallel to the load, starting from the outer layers. Consistent with the observation by Gao et al. [5], first transverse matrix cracks appear at 0.25-0.45% strain level, although that is a strain level much lower than the failure one. In Fig. 13a, the red arrows indicate 2 cracks propagating in the transverse fill; a crack density of 0.2 cracks/mm has been detected. Transverse cracks, also near the PET film are depicted in Fig.13(b) that consider an applied strain level of about 0.65%.



(a) (b)

Figure 13: Cracks and delamination between CFRP and PET (a) and cracks and PET failure (b).

It was observed that numerous cracks start from regions with matrix cracks in the tows. Since the interstitial regions are usually resin-rich, cracks and delamination often initiate from these sites and extend to fill tows. Once the crack has reached the dielectric film surface two different phenomena may occur: the crack start to run parallel to the dielectric film (delamination, see fig.13a), or it propagate through the PET film causing the local failure of the dielectric (see fig.13b).

4.5 Out-of-plane tensile test

As it is expected, the capacitors analyzed exhibits a very low out-of-plane tensile strength. All specimens tested have shown a failure at the PET/CFRP interface. The image of a specimen after the failure is shown in Fig. 14.

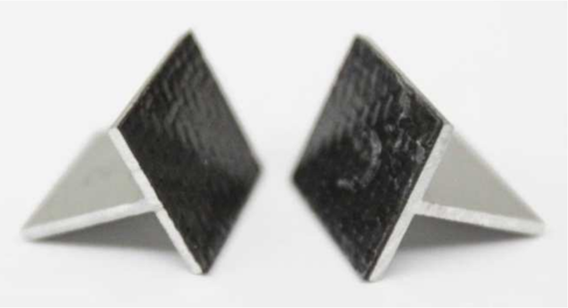


Figure 14: Image of an out-of-plane tensile specimen after the adhesive failure.

Specimens failed under a load of 365÷392 N, which correspond to a strength of about 1 MPa.

5. INTERLAMINAR STRESS ANALYSIS AND FAILURE LOAD PREDICTION

In previous tensile tests performed on CFRP structural capacitors, it was observed that a premature material failure is mainly due to delamination between the thin dielectric PET film and the CFRP layers. This is caused by the interlaminar stresses at the free-edges, due to the elastic properties mismatch.

In order to detect the actual interlaminar stresses as well as to implement a reliable criterion to predict the strength of the analyzed capacitors, FEM simulation has been performed by using ANSYS code.

5.1 FEM analysis

A finite element simulation of the tensile test has been performed to detect the corresponding stress field at the interface between the PET film and the adjacent CFRP layers. Fig. 15 shows the σ_z and τ_{yz} distributions along the interface between PET and CFRP.

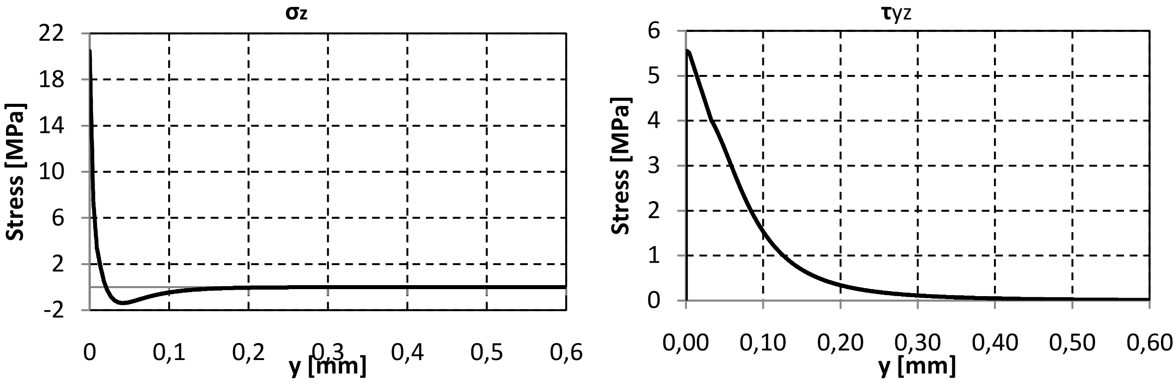


Figure 15: σ_z and τ_{yz} distribution at the interface between PET film and adiacent CFRP layers.

From fig.15 it is clearly seen that both the interlaminar stresses show a typical singular trend at the free-edge.

5.2 Failure criteria

Since the experimental analysis has shown that the failure is due to delamination phenomena at the CFRP laminate/PET interface, a reliable failure criterion that can be used to predict the ultimate load, has to consider the high interlaminar stress gradient near the free edges.

In other words, a delamination criterion has to be associated with a proper approach that consider the actual stress gradient and the local damage tolerance of the material. Criteria that satisfy such an approach are the so called Point Stress Criterion (PSC) and the Average Stress Criterion (ASC).
Because the non-monotonic trend of the σ_z interlaminar stress, the ASC has been chosen and the corresponding characteristic distance y_0 has been experimentally computed by applying the relationship [8]:

$$\bar{\sigma}_{ij} = \frac{1}{y_0}\int_0^{y_0} \left|\sigma_{ij}\right| dy \qquad (1)$$

In detail, the failure condition is determined by using the formula introduced by Marion [10], i.e.:

$$\left(\frac{\bar{\tau}_{yz}}{\tau_{yz}^{F}}\right)^2 + \frac{\bar{\sigma}_z}{\sigma_z^{TF}} = 1 \qquad (2)$$

In eq.2 the failure shear stress τ_{yz}^{F} coincides with the shear strength obtained by the ILSS test, whereas the failure tensile stress σ_z^{TF} is the value determined by the out-of-plane tensile test. $y_0 = 64$ µm has been obtained by applying the proposed method to the experimental tensile test. Such a value is very close to the total thickness of the dielectric layer.

5.3 Application of the proposed method to the specimens with angled edge

As described in chapter 3, to avoid short circuit between layers, all the tensile test specimen have been manufactured with angled edges. The experimental evidence has shown that, although the angled edges allow to avoid short circuits between opposite CFRP layers, they do not influence the mechanical strength of the specimens. Such a result have already been obtained in [9], by using proper 3D singular wedge finite elements. In this section, in order to assess the reliability of the strength criterion proposed in the previous chapter, it is applied to the estimation of the failure load of CFRP capacitors having various angled edges (0°, 20° and 45°). Such estimation have been carried out by considering the interlaminar stress distribution obtained by FEM simulations. For each case examined the corresponding values of the average interlaminar stresses and the value of (eq.2), are reported in Table 5.

Table 5: Average interlaminar stresses and failure prediction obtained by using the proposed criterion.

Edge angle	σ_z [MPa]	τ_{yz} [MPa]	Proposed criterion
0°	0.890	4.100	1,000
20°	0.874	3.697	0,983
45°	0.875	3.091	0,980

Finally, the results reported in Table 5 shows that, although the presence of angled edge reduces significantly the stresses singularity order, in accordance to the experimental evidence the characteristic value of the failure parameter is still close to the failure value, i.e. to 1. Consequently, it is possible to state that the proposed strength criterion can be used for reliable estimation of the mechanical strength of the analyzed CFRP capacitors, under a generic load condition.

6. CONCLUSIONS

This paper presents a systematic study on the effects of the applied mechanical load and damage on the electrical performance of CFRP structural capacitors. To this purpose a proper test method to characterize the effect of mechanical load and distributed damage on the capacitance of structural capacitors, was developed and employed. In addition, to increase the mechanical strength, the dielectric polymer film was treated with a NaOH.

Tensile tests carried out on charged structural capacitors specimens subjected to strain levels of about 0.6-0.65%, have shown a drop of capacitance of about 50% due to significant damaging of both the CFRP layers and the PET film. Such a damage have been confirmed by a proper fractographic analyses, that have shown the presence of matrix cracks and delaminations; in general a matrix crack density of 0.2 cracks/mm have been detected.

Moreover, after unloading the capacitance is recovered, and in many cases also improved. Furthermore, an out-of-plane tensile test has been designed and performed to evaluate the out-of-plane strength strictly related to the adhesion between CFRP layer and PET film.

Finally, in order to implement a failure criterion that can be used at the design stage for the strength prediction of the analyzed capacitors under various load conditions, finite element analyses of the interlaminar stress distribution in the tensile test, have been carried out by using proper numerical models and discretizations. Due to the significant mismatch of the elastic properties of PET and CFRP layers, typical singular trends for σ_z normal stress and the τ_{yz} shear stress are found; according with the failure criterion adopted, they lead to significant delamination phenomena at the CFRP/PET interface. Moreover, according with the experimental evidence, the application of the proposed failure criterion to various CFRP capacitors with angled edges, has permitted to highlight that, although the singularity order decreases, the angled edges do not have any influence on the failure load.

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