INFLUENCE OF THERMAL CYCLING ON THE LOW VELOCITY IMPACT RESPONSE OF CFRP LAMINATES

D. Ghelli[a*], V. Cantarini[b], E. Troiani[b]

[a]*Interdepartmental Center for Industrial Research on Advanced Mechanics and Materials (CIRI-MAM), University of Bologna, via Umberto Terracini 24, 40131 Bologna - Italy*
[b]*Department of Industrial Engineering (DIN) - University of Bologna, Forlì Campus, via Fontanelle 40 - 47121 Forlì - Italy*
**daniele.ghelli@unibo.it*

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Abstract
Low velocity impact tests were carried out on unidirectional CFRP laminates previously aged with up to 80 thermal cycles between temperatures of -16 °C/+80 °C, in order to assess the effect of repeated temperature changes on the impact behavior of the material. The specimens subjected to the highest numbers of thermal cycles present a lower energy absorption during impact. In addition, the load drops that are usually visible in the contact force history of impacted laminates, and are attributed to the propagation of delamination, tend to disappear as the number of thermal cycles increases. On the other hand, both visual inspection and optical microscope analysis showed that no significant difference is induced by thermal cycling in the extent of delaminations. Thermal aging probably induced an embrittlement in the material, due to matrix microcracks that are generated at the lowest temperatures.

1. Introduction

Carbon Fiber Reinforced Polymers (CFRP) are widely appreciated in every field where lightness, together with high resistance and stiffness, are required. Moreover, CFRP present other characteristics, like low thermal and electrical conductivity, flame retarding ability, corrosion resistance, that make them useful for many specific applications. The first field in which they were used has been civil and military aeronautics and space industry, where nowadays composites represent one of the main materials employed in the structural parts of aircrafts. Among the concerns regarding practical use of CFRP, their resistance to impact of foreign objects is one of the most important. Their mechanical behavior is essentially brittle, therefore when they are subjected to collision they absorb impact energy mainly through internal damage, that can be in the form of matrix cracking, delamination and fiber fracture. The unique externally visible damage is the indentation created by the impacting object. Every other type of damage can be barely visible externally or not visible at all. At the same time it can cause an important reduction of the mechanical properties of the material. Since collisions of external bodies are common in the lifetime of a component, the vulnerability of CFRP to impact can be considered one of the most important research themes regarding these materials [1].
CFRP are also more sensitive than other materials to environmental conditions, in particular to extreme temperature variations (very high or very low temperatures). Some information

about the problems that these materials present at low temperatures can be found in the literature regarding applications of composites in arctic environment. It is known that residual stresses are generated by the curing process and can be amplified at low temperatures, producing matrix microcracks [2]. It is possible that these microcracks represent starting points of delaminations when the material undergoes external mechanical loading. Moreover, when the material is subjected to repeated temperature variations the microcracks induced by residual stresses can propagate. Therefore if the material undergoes thermal cycling, then it is possible that its mechanical properties are degraded.

Since the effect of thermal cycles is an alteration of the physical and chemical properties of the material, as a consequence the dynamic response and the damage due to impact can be influenced. In other words, an interaction between impact response and environmental conditioning of the material can exist, thus impact resistance can be modified in thermally aged laminates. This is the justification for the interest about this issue. Indeed in many applications composite materials are subjected to thermal cycles during their lifetime. For example, the fuselage panels of airplanes that fly along arctic routes endure very low temperatures. In this way cracks can be induced, especially in the proximity of junctures or other parts withstanding high stresses. Conversely, ultralight aircrafts that travel between isles of tropical seas may present structural damage due to high temperatures.

In the literature a very small number of contributions can be found about the effect of thermal cycles for long periods on the impact behavior of polymeric matrix composites. No previous study is available especially regarding unidirectional CFRP. All published researches focus on other polymeric composites (like glass fiber laminates) or present the results of impact tests followed by thermal cycling (shorter or longer), or investigate the effect of abrupt temperature changes imposed only once to the material.

Several authors [2-6] study the effects of thermal cycles on the mechanical properties of polymeric matrix composite materials. Dutta [3] in 1988 performed mechanical tests on glass/epoxy laminates after thermal cycles between +60 °C and -60 °C, demonstrating that the material strength is reduced as the number of thermal cycles increases. Kasap et al. [4] study the effect of thermal cycling at high temperature on the flexural strength of three different types of E glass fiber composites. They observe a reduction of the flexural strength and stiffness when the number of thermal cycles becomes higher, and attribute this effect to fiber-matrix debonding. Hosur et al. [5] subjected woven carbon/epoxy laminates to low velocity impact tests followed by cold-dry and cold-moist thermal cycles for a period of 3 and 6 months. They observe that the specimens aged for the longest period present a wider damage extent. Dutta and Hui [2] carried out thermal cycles between temperatures of +50 °C and -60 °C on glass fiber composites of two different types. They find that the Young and shear moduli are reduced by thermal aging, and think that this effect is due to microcracking of the polymeric matrix. Tai et al. [6] carried out low energy impact tests and fatigue tests on unidirectional carbon/PEEK laminates previously subjected to thermal cycles between +60 °C and -60 °C. According to their conclusions, thermal aging had no effect on the impact response of the material, because the matrix did not present microcracking as a consequence of thermal cycles.

It appears clearly from the analysis of previous literature on the subject that very limited knowledge is available regarding the effects of thermal aging on the impact response of unidirectional CFRP. On the other hand, we think that such knowledge is of main importance for a number of technical applications of CFRP in many different fields, including aeronautics, civil constructions in arctic or tropical environments and marine engineering. To the authors' knowledge, the present study is the first specifically focused on the low velocity impact behavior of unidirectional carbon fiber laminates previously subjected to thermal cycles. 35 specimens divided in 7 groups, each one exposed to a different number of thermal cycles (up to a maximum of 80) between the temperatures of -16 °C/+80 °C, underwent a low velocity impact of approximately 6 J. Representative specimens were sectioned and observed by an optical microscope to assess damage types and extent. Results, in terms of contact force history and energy absorption, are presented and discussed.

2. Materials and experimental procedure

The specimens used in experimental tests were obtained by the hand lay-up technique starting from a roll of unidirectional carbon fiber/epoxy resin pre-preg. 12 layers of this material were placed in a suitably designed and manufactured die. According to the stacking sequence $[0/90/0/90/0/90]_s$, the pre-preg layers were cut in 12 rectangular sheets for each laminate, 450 mm long and 400 mm wide. In this way we obtained 4 panels, each one consisting of 12 layers, with an average overall thickness of 1.7 mm. Then these panels were cured in autoclave following the temperature and pressure cycle recommended by the manufacturer. Rectangular specimens 150 by 100 mm were cut by means of a diamond saw. These dimensions are recommended by the ASTM code D 7136 [7], regarding low-velocity impact testing of composite laminates. 35 specimens (7 groups of 5 samples) were used for the present study. Each of these groups underwent a different number of thermal cycles. The first group, named A, was not subjected to any thermal conditioning. The following groups, named B through G, were subjected to increasing number of thermal cycles: 1 cycle (group B), 3 cycles (C), 12 cycles (D), 24 cycles (E), 48 cycles (F), 80 cycles (G). In all cases each thermal cycle lasted 24 hours: 12 hours in oven whose temperature was kept constantly at +80° C and the following 12 hours in a freezer at the constant temperature of -16 °C. In order to avoid direct exposure of the specimens to abrupt temperature changes they were protected by a thin aluminum sheet, both in oven and in freezer.

After thermal cycling all the 35 specimens were subjected to low velocity impact test carried out in a drop-weight tower. In every test the drop height was 0.5 m and the impactor mass was 1.25 kg. Therefore the nominal impact energy was 6.1 J. During the impact test the specimen was placed on the support fixture of the machine and held in position by four lever clamps with rubber tips, according to the norm ASTM D 7136 [7]. The support fixture had a rectangular opening 125 by 75 mm. The specimen was exactly centered with respect to this opening and the impactor hit the specimen exactly at its center. Multiple impacts were prevented by stopping the impactor manually just after the first contact with laminate. After impact all the specimens were subjected to visual inspection in order to detect external damage. One specimen was selected in each of the groups A through G for detailed inspection by means of an optical microscope. To this end, the selected coupons were incorporated into epoxy resin, then sectioned along the main axis of the laminate and finally polished. Then the laminate section was observed with the microscope in order to detect internal damage.

The impactor was equipped with a piezoelectric load cell, that is the unique part of the impactor directly in contact with the laminate during the impact test. In this way a precise measure of the contact force as a function of time was obtained. The laser device fixed to the support plate of the drop-weight tower provided a measure of the actual velocity of the falling impactor. Both the force and the laser signal were acquired at sampling frequency of 100 kHz, without any filtering except the intrinsic one due to the measurement chain. More details about the drop-weight tower can be found in [8]. The force signal was numerically integrated twice according to the procedure suggested by ASTM D 7136 norm [7], in order to obtain velocity and position of the impactor as a function of time. Laser data were used to provide the initial conditions for the integrations. In accordance with the definition in ASTM D 7136 code, absorbed energy E_a at the time t was calculated according to the following formula:

$$E_a(t) = m\frac{v_i^2 - v(t)^2}{2} + m\,g\,\delta(t) \tag{1}$$

In (1) v_i is the initial velocity of the impactor, $v(t)$ is the velocity of the impactor at the time t, g is acceleration due to gravity, m is the impactor mass and $\delta(t)$ is the impactor position at the time t (positive downwards, measured from the impacted surface of specimen).

3. Experimental results and discussion

Figure 1 presents the contact force history recorded during impact tests of some representative specimen groups. As it is shown in this figure, the laminate dynamic response to impact is dominated by the first vibration mode and the higher modes give a little contribution to the dynamic behavior. Indeed the contact force-time curve is similar to a half-sine wave with smaller waves with higher frequency superposed to it. In particular the effect of the higher modes can be seen in the initial part of graphic, where the first of this smaller waves appears in all plots [1]. In general graphics of all groups show that the 5 tests carried out of each group gave similar results. In particular this can be seen in the first part of the plots of contact force history where it is possible to see a similar behavior in spite of small thickness differences among specimens. In many cases several load drops are visible in the plots, that are usually attributed to the propagation of delamination. Some degree of variability can be seen within each group regarding the amplitude of such load drops. Nevertheless, the amplitude of drops clearly tends to decrease as the number of thermal cycles increases. Generally, the visual inspection of specimens has shown that the delamination is clearly visible in the form of a little swelling on the un-impacted face of the laminate and has a similar dimension in all specimens. On the other hand the laminate impacted side presents small dent, fiber micro-buckling close to the dent and matrix splitting.

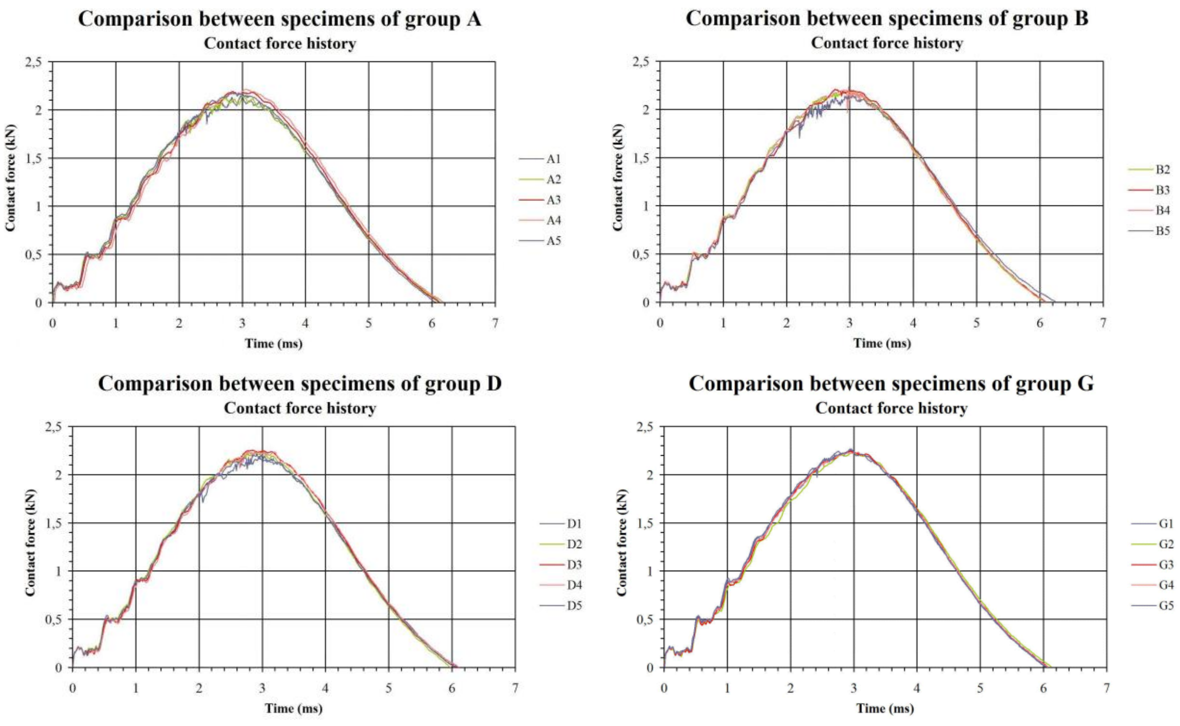


Figure 1. Representative contact force history recorded during impact tests of all specimens of groups A, B, D and G. The plot of the specimen B1 is missing because there has been a problem in data acquisition system.

The delamination size, as it can be appreciated by visual inspection, was consistently confirmed by microscope analysis of sectioned laminates. As shown by Figure 2, delaminations are present at each interface between a 0° ply and the adjacent 90° ply located above it. In every case the widest delamination is found at the lowest interface, above the 0° external ply on the un-impacted face of the laminate. The length of the swelling on the laminate back face corresponds to the length of this delamination as can be measured by microscope observation. As can be seen in Figures 3-5, no definite effect of the different thermal aging can be recognized neither in the extent of the largest delamination (Figure 3) nor in the sum of the lengths of all delaminations (Figure 4). The measure of each of the single delamination observed at all interfaces, reported in Figure 5, again indicates that the extent of this damage type appears not to be affected by the number of thermal cycles.

The variability from one specimen to another, visible from the differences both in the contact force history and in the delamination size, did not show any correlation with the variability of the specimen thickness. Therefore some of these differences can be likely attributed to thermal aging. We suppose that the different behavior of specimens can be due to a dissimilar exposure to the temperature changes during the thermal cycling. It is possible that depending on the position of specimens in oven and in freezer, the time taken to reach the extreme temperatures of the cycles was not the same for every sample. It is known that low temperatures can have a detrimental effect on the matrix, that can influence the response after impact, producing internal damage in the material [3]. Therefore it is possible that the specimens that experienced a more rapid variation of temperature underwent more severe damage.

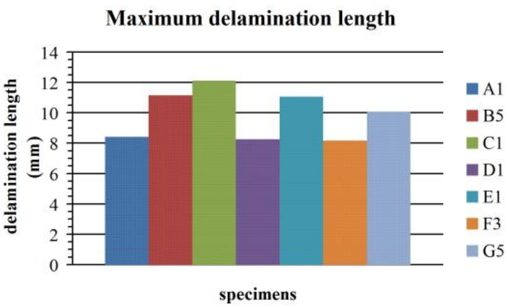
Before starting to analyze the experimental results we have proceeded to exclude any possible cause of systematic error. Because in the following we will discuss on the energy absorbed by the specimens during impact, in the first time we want to exclude the possible sources of error influencing this parameter. The first is the energy absorption due to the indentation of laminate by the impactor head. Our test conditions were low velocity, low energy impact (6.1 J) and thin specimens, that undergo very large deflection. Therefore the dent generated by the impactor was very small and the energy spent in creating the dent is negligible with respect to the impact energy [1]. As a consequence possible variation of this energy can be disregarded. Another possible cause of systematic error on energy absorption was the kinetic energy stored in the specimen after the impact. But also this energy is negligible with respect to the initial kinetic energy of the impactor. Indeed in the low velocity impact the dynamic response can be considered with a good approximation as a sequence of equilibrium states [1].

Another possible cause of error may have been the actual velocity of impact on the specimens. As shown by the Figure 6 the average velocities measured for each specimen group are slightly different. In the specimens of groups from A to E the average velocity increased in consequence to decrease of friction between the impactor and the rails of drop weight tower as the test program proceeded. The same effect is noticed on the specimens of the groups F and G, that were tested subsequently. On the other hand these differences of velocities are numerically small (less than 2% of the average value). In addition the trend shown in Figure 6 is completely different from the one in Figure 7 regarding the absorbed energy. Therefore we could not recognize any relationship between the absorbed energy and the impact velocities. The last parameter that could have influenced the experimental measure of the absorbed energy is the specimen thickness (which appears elevated to the third power in the expression of the flexural stiffness of the laminate). The thicknesses of the individual specimens are slightly different from each other. Nevertheless observing the data of single tests no definite relationship between absorbed energy and laminate thickness can be seen.

Having discussed and excluded all possible causes of systematic error, we can conclude that the differences observed between the energy absorption in the sample groups can be attributed to the dissimilarities in the damage caused by impact.

Figure 2. Optical microscope photographs of specimens A1, B5, C1, D1, E1, F3, G5 (from top to bottom) sectioned along the 0° direction through the impact point. The impacted face of laminates is on the low side of each picture.

As it is known [1] the principal types of damage observed in the low velocity impact of composite laminates are matrix cracking, delamination and fiber fracture. Regarding matrix cracking (that is also visible on our specimens), the previous studies on this topic suggest that this damage mode is responsible for the smallest energy absorption. It also is the first mode of damage appearing even at the very low impact energy levels.



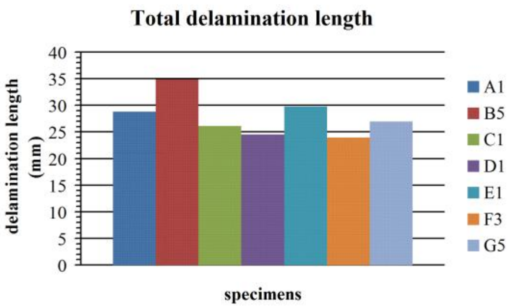


Figure 3. *(left)* Length of the largest delamination measured in sectioned specimens.

Figure 4. *(right)* Sum of the delamination lengths observed at all interfaces in sectioned specimens.

Generally on the other hand fiber fracture requires a very high level of energy to be generated. But in our tests the impact energy was limited, because the drop height was small (0.5 m). For this reason, as previously noted, only a few specimens present fiber fracture of little extent. Microscope photographs (Figure 2) also show that fiber rupture is not significant in the present tests. Then in our case we can suppose that the energy absorbed by fiber rupture was negligible with respect to the total absorbed energy.

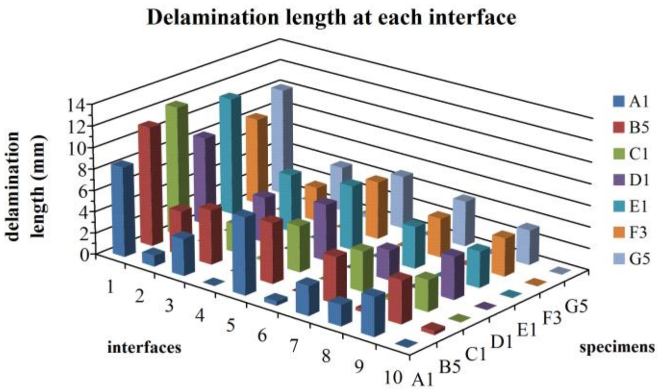
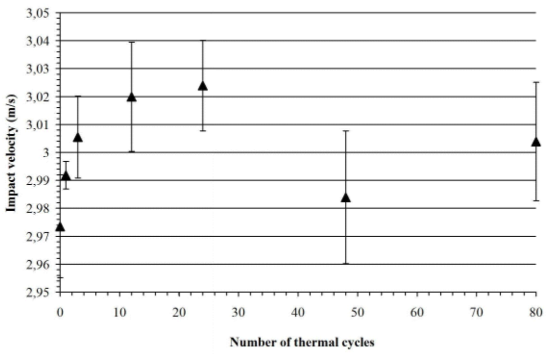


Figure 5. Single delamination lengths measured at all interfaces in sectioned specimens. Interfaces between plies with different fiber orientation are numbered starting from the lowest one (just above the 0° layer on the un-impacted face).



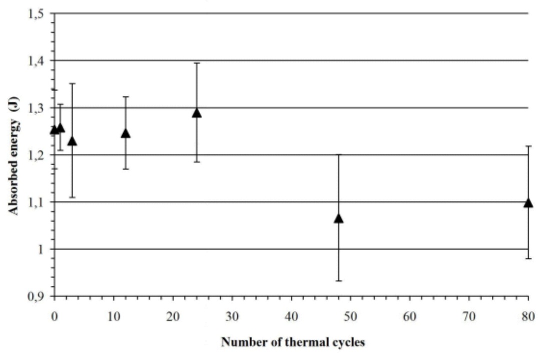


Figure 6. Average values of measured impact velocity for each specimen group, as a function of the number of thermal cycles. Error bars indicate the standard deviation.

Figure 7. Average values of calculated absorbed energy during impact, for each specimen group, as a function of the number of thermal cycles. Error bars indicate the standard deviation.

Therefore the main type of damage in the present tests was delamination, that can be considered responsible for most part of energy absorption and load drops observed in the contact force history. The samples of the groups F and G present lower absorbed energy (see Figure 7). In principle, this result may be interpreted in two different ways: the first possible explanation may be that a smaller fracture surface was created. Another interpretation can be that a smaller quantity of energy was necessary to create a similar delamination surface. Both visual inspection and microscopic observation, showing that no significant difference exists between delaminated areas of different specimen groups, support the second hypothesis. This means that a smaller amount of energy was spent to create the same surface of fracture in terms of delamination, consequently the behavior of the material was more brittle.
The contact force-time curves (Figure 1) show that an increasing number of specimens do not present load drops with the increase of thermal cycles number. This suggests that the propagation of delamination occurred in a gradual, stable way, rather than suddenly. Because in literature it is possible to find that the thermal cycles produce microcracks in the material [3], then it can be supposed that the propagation was more gradual due to the presence of some microcracks, that acted as a trigger for the delamination onset. Microscope analysis of the polished laminate section away from the impact point (where one can expect that only damage generated by thermal aging can exist) did not display any matrix crack. On the other

hand, actual presence of cracks can not be excluded. Possible cracks may be left undetected at a certain distance from the impact point, where permanent deformation of the specimen after impact is negligible, thus cracks are not kept open by ply bending. Indeed, as can be seen in Figure 2, matrix cracking is visible in the vicinity of the dent induced by the impactor head, where local strain is much larger.

4. Conclusions

Low velocity impact tests were carried out on unidirectional carbon fiber/epoxy resin laminates, previously subjected to various numbers of thermal cycles between the temperatures -16°C/+80°C. All the specimens were visually inspected after impact. Selected specimens were sectioned and observed by optical microscope. The data recorded by the drop weight machine were analyzed.
The analysis of experimental results led to the following conclusions.
1) The specimens that were subjected to a higher number of thermal cycles (48 and 80 cycles) showed a lower energy absorption. On the other hand they presented delamination of similar extent to the one noticed in the other specimens.
2) By the observation of the impact response of laminates in terms of contact force history load drops can be recognized that are usually attributed to the propagation of delamination. These load drops however tend to disappear as the number of thermal cycles increases.
3) Thermal aging induced a modification in the mechanical behavior of material making it more brittle. We can suppose that thermal cycles produced microcracks in the matrix. Such microcracks can act as starting points of delamination and their number increases with the number of thermal cycles. This can explain why the propagation of delamination is more stable in the specimens that underwent the longest aging as shown by the almost total absence of load drops in the contact force history.

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