MICRO-CT ANALISYS OF INTERLAMINAR GRADED INTERFACE STRENGTH (IGIS) COMPOSITES BASED ON A THERMOPLASTIC MATRIX

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Abstract

The hybridization approach in composites is used to improve the impact resistance and the damage tolerance of laminates. It is usually based on the use of two different reinforcing fibres (alternatively stacked, commingled or interpenetrated), one having high stiffness and the other having high toughness. Such approach is effective in improving the low velocity impact resistance of the composites but it poses new issues related to the different specific properties of used fibres (coefficient of thermal expansion, interface compatibility, residual stresses after the laminate production to name a few). A new design has been recently proposed for thermoplastic composites based on the gradation of the interlaminar interface strength (IGIS). The interface strength between fibres and matrix is graded through the thickness by alternating woven fibres with compatibilized or not compatibilized polymeric layers. IGIS laminates involving a commercial grade of polypropylene (PP) as matrix and a woven glass fabric as the reinforcement have been prepared with different interface strength configurations (symmetrically or asymmetrically with respect to the middle plane) by means of the well-known film stacking technique. In this contribution Micro Computerized Axial Tomography (MicroCT) analysis was used to investigate the actual damage mechanisms occurring in IGIS laminates and showed how the graded interface strength is capable of shifting fibre breakage at higher impact energies and allowing elastic recovery in conditions such to significantly damage the fully compatibilized laminate configuration.

1. Introduction

Recently, a growing interest has been devoted to the development and production of cost effective fibre reinforced thermoplastic polymer composites. Thermoplastic composites, often processed by stacking alternating layers of fibres and polymer sheets in a hot press, offer superior handling and formability. The benefits undoubtedly include a low cost per part, short processing times and potential recyclability of products. Other advantages, such as the improved fracture toughness, high damage tolerance and good resistance to micro cracking, the possibility to re-shape or re-mould the laminates at high temperatures, lead to relevant perspectives in many industrial fields [1].
The polypropylene/glass fibres system has been thoroughly investigated under different conditions [2]. Polypropylene is widely used for its low cost, high specific performances and well assessed recycling process. Glass fibres (GFs) offer the same high performance/cost ratio, coupled with a high chemical resistance and fairly good mechanical properties. GFs have a lower elastic modulus with respect to carbon fibres, a lower resistance to fatigue and usually show poor adhesion at the fibre-matrix

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interface, leading to a significant decrease in the static mechanical properties [3, 4]. To enhance performances, the stress transfer through the fibre/matrix interface region must be improved. This issue can be faced by physical pre-treatments of fibres' surface [4, 5] and/or by using suitable coupling agents, like maleic anhydride, to enhance the wetting of the hosting matrix on fibres [3-5]. Composite laminates are very prone to impact damaging, which significantly reduces the in service structural properties, and show a brittle behaviour during impact that limits the dissipated energy [6]. A growing concern in structural applications are the low velocity impacts, representative of real events occurring during fabrication, maintenance and service of such composite systems [7, 8]. Various approaches have been used to enhance the impact damage resistance of composite laminates. Among these, it is worth to mention the hybridization. The inclusion of high strain-to-failure fibres in the composite configuration has been demonstrated to be a very effective way to improve the impact energy absorbing capability [9]. In fact, fibre hybrid laminated composites combine the good static performances of highly stiff fibres with the excellent impact resistance of ductile ones [10] but at the expenses of the reinforcement balance and symmetry with respect to the middle plane.

Tomography (or Computerized Axial Tomography, CT) is a non-destructive technique for the study of the physics and mechanics of materials, given its great interest for the analysis of complete volumes. The basis of this technique is the reconstruction of three-dimensional volumes from a sequence of projection images (generally obtained from an exposition to an X-Ray beam) taken at different equally spaced angles around the object. The result of the reconstruction is a series of images (slices) at different consecutive sections along one or more axis. The reconstruction of the X-Ray projections provides a three-dimensional spatial distribution (3D map) of the X-Ray absorption capability of the material. Different phases (or characteristics) of a material, having different absorption capacities, can be easily identified by means of the mentioned three-dimensional map.

In this work, we analysed the effects of impacts performed ad different impact energies on the microstructure of IGIS structures (a new class of hybrid composites in which the hybridization concerns modifications of the matrix rather than of the reinforcement). The use of laminates with different interlaminar interface strengths allows both high impact performances, coming from the presence of low interacting matrix and reinforcement, and high static mechanical properties, typical of composites based on compatibilized matrix [11-13].

2. Experimental

The matrix was a polypropylene (PP) resin supplied under the trade code MA712 by Unipetrol (Czech Republic; MFI = 12 g/10 min). A maleic anhydride grafted polypropylene (PPgMA) was used at 2.0 % by weight to improve the polymer/fibres interface (Polybond 3200 from Chemtura, Philadelphia - PA, USA; MFI 115 g/10 min, 1 wt% maleic anhydride content). A plain weave type glass woven fabric (E-type glass fibres having density of 2.54 g/cm^3, functionalized by amino silane groups) with a specific mass of 204 g/m^2 was considered as reinforcement.

Composite laminates were produced by using the film stacking technique and a compression moulding machine (model P300P, Collin GmbH, Ebersberg, Germany). Neat or compatibilized polypropylene film layers were alternatively stacked between 20 layers of glass woven fabrics, and compression moulded according to suitable temperature and pressure profiles [11, 12]. IGIS configurations were prepared by changing the stacking sequence of compatibilized and not compatibilized PP as reported in Table 1 [13]. Fabric layers were kept balanced in all laminates using a 0°/90° symmetric arrangement with respect to the middle plane of the laminate with a target thickness of 3.3 mm and a target glass fibre content of 45% by volume. Further details on the laminates and the production process are reported in [13].

Impregnation, voids content and flexural properties are reported in [13]. Low-velocity impact tests were performed at impact energies (E_i) of 6 J (to induce a limited damage) and 27 J (representing the threshold value (Peak value) for inducing extensive fibre breakage in the fully compatibilized laminate). An instrumented drop-weight impact testing machine (model Fractovis Plus from CEAST, Pianezza (TO), Italy) equipped with a hemispherical tip (diameter 12.7 mm) was used. The impact velocity was kept constant and equal to 2.5 m/s. The sample holder was a stainless steel annular ring (internal diameter 40 mm, outer diameter 60 mm). Tomographs acquisitions and 3D reconstructions

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have been performed at PPRIME Institute (Poitiers, France) using an UltraTom CT scanner manufactured by RX Solutions. A 16 µm resolution was applied.

Table 1. Coding of IGIS laminates and layer sequences (N = not compatibilized PP layer, C = compatibilized PP layer).

Sample	*Polymeric layer sequence*
NEAT	20 N
COMP	20 C
HNC	10 N/10 C
HCN	10 C/10 N
HNCN	5 N/10 C/5 N
HCNC	5 C/10 N/5 C
HX	2 C/5 N/6 C/5 N/2 C

3. Results and discussion

Laminates showed no voids after several optical acquisitions. Tests performed with digestion methodology resulted in void volume fraction well below the required 2% limit of acceptance. The composite prepared with not compatibilized polymeric layers (NEAT) showed very weak interface strength. The poor fibre/polymer interface allowed the slippage of fibres during the laminate deflection (Figure 1A). On the contrary, in fully compatibilized laminates (COMP) fibres are very well wetted and the matrix clearly adheres to the fibres surface (Figure 1B). These opposite behaviours are visible in IGIS laminates, where characteristic fracture surfaces of neat and compatibilized layers can be identified and associated to the stacking sequence [11].

Flexural tests always led to the breakage of the compression side of the sample. The marked damaging of the composite in the upper side comes from the compressive failure of glass fibres and occurred at a lower stress in non compatibilized composites with respect to compatibilized ones [12, 13]. Beyond the peak stress, a lowering of the stress-strain curve was detected in all laminates due to the propagation through the laminate thickness of both interlaminar and intralaminar damaging until the back side is reached.

The presence of the coupling agent had a positive effect on both flexural modulus and flexural strength [13]. The COMP laminate clearly showed improved performances with respect to the NEAT one. In particular, the flexural strength resulted to be strongly increased in presence of the coupling agent. This result comes from the enhanced capability, in compatibilized systems, of better transferring the load between the matrix and the woven fabric through the interface.

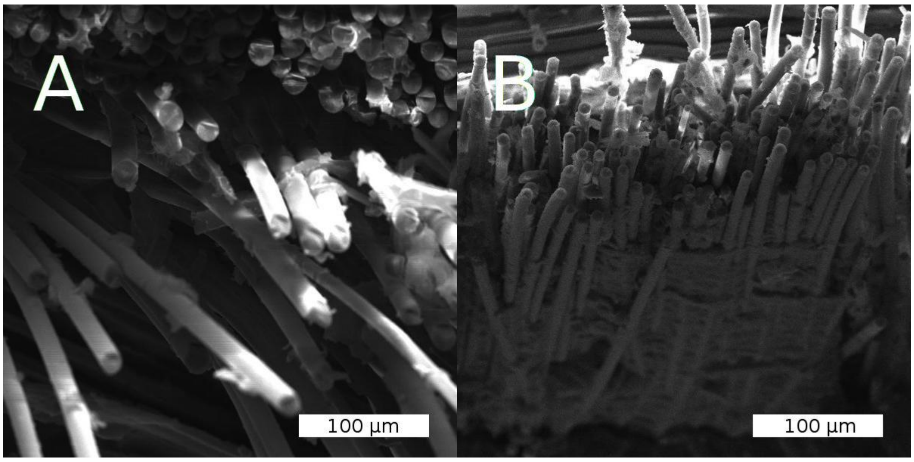

Figure 1. Fibre wetting in NEAT (A) and COMP (B) laminates.

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IGIS composites showed flexural properties in between those exhibited by COMP and NEAT systems. Since the total amount of compatibilized and not compatibilized layers was the same in all configurations, the position and sequence with respect to the middle plane was responsible for the different stiffness and strength of the laminate [13].

The low velocity impact characterization of NEAT and COMP laminates showed a different behaviour with respect to the static bending test. At low impact energy (E_i = 6 J) the COMP laminate showed a higher peak load and a higher recovered energy with respect to NEAT laminate in accordance with the higher stiffness of the composite structure. The compatibilized laminate incurred in lower damaging. IGIS laminates showed intermediate results under the same impact conditions. The highest peak displacement, which is the maximum deflection of the laminate during the impact event, was exhibited by the NEAT laminate. Almost all IGIS configurations behaved as the COMP composite in terms of peak and residual displacements, showing comparable external damages [13].

As the impact energy rose to E_i = 27 J, which represents the peak impact energy for the COMP laminate, the NEAT system behaved better than the COMP one exhibiting higher values of both peak load and recovered energy. It is evident from the shape of the Force vs Displacement curve that at E_i = 27 J the COMP configuration incurred in fibre breakage since a 4.2 mm in displacement (displacement at peak load) is markedly different from what happens for the NEAT laminate (Figure 2). Moreover, the higher peak displacement confirmed its lower strength and capability to recover elastic energy. At E_i = 27 J relevant differences were detected in IGIS laminates but all configurations showed a performance better than the one exhibited by the COMP laminate.

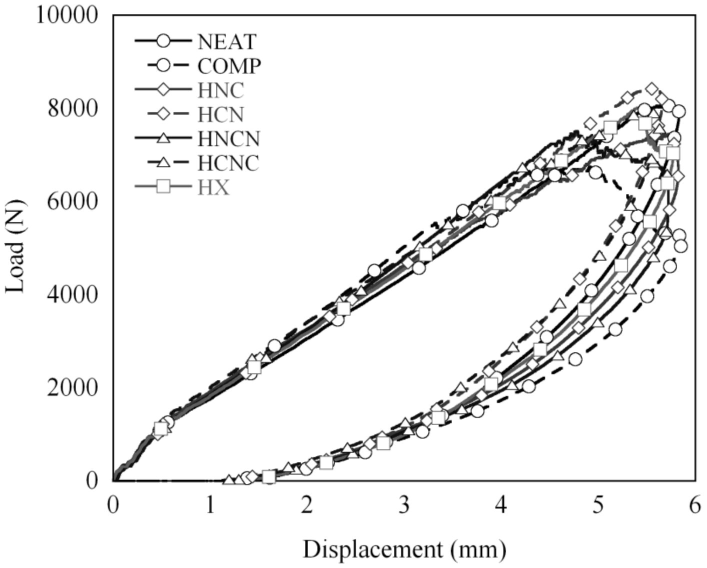

Figure 2. Load versus Displacement plots of a 27 J-impact on the investigated laminates [Reported from 13]

HNC, HNCN and HX samples showed peak displacements comparable to the COMP ones but higher recovered energies while HCN and HCNC laminates showed the lowest peak displacements and the highest recovered energies. More interestingly, Load versus Displacement curves did not show any apparent signal of crucial fibre breakage. These latter two IGIS configurations exhibited a sound improvement over the compatibilized laminate impact performances and were able to preserve the integrity of reinforcing fibres.

This consideration was supported by the microCT analysis. The comparison among COMP, NEAT, HNC, HCN, HCNC and HX laminates impacted at 27 J is shown in Fig. 3. It is evident that the COMP laminate has been severely damaged at 27 J, and a crack propagating through the thickness was generated. On the contrary, the NEAT laminate, in which all layers were not compatibilized, showed

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only an early breakage of fibres in the outer three layers on the back side of the sample. IGIS structures exhibited an impact behavior dependent on the compatibilized/not compatibilized sequence. In particular, the impact damage appeared to be proportional to the amount of compatibilized layers of the back side of impacted laminates. The most damaged IGIS configuration was the HNC, which is characterized by the upper half (impacted side) of the laminate made of NEAT layers. HNC exhibited a complete breakage of the lower half (made of compatibilized matrix) of the laminate (Fig. 3C). On the contrary, impacting the same IGIS configuration from the C side (HCN sample, Fig. 3D), a very limited damaging occurred with fibre breakage confined in the three outer layers on the back side (in the same fashion as the NEAT laminate in Fig. 3B). The HCNC laminate (Fig. 3E), constituted by a smaller number of compatibilized layers with respect to the HNC sample, showed a lower damaging in relation to HNC (five broken layers) but higher than that of the HCN laminate. The presence of the not compatibilized layers stopped the crack propagation, preserving the remaining part of the laminate and, in turn, the capability of the laminate to bear loads. The effectiveness of the IGIS configuration is particularly evident in Fig. 3F, where the section of the HX laminate is shown. Such composite was characterized by the breakage of three outer layers in the back side, in accordance with the interface strength gradation configuration (Table 1).

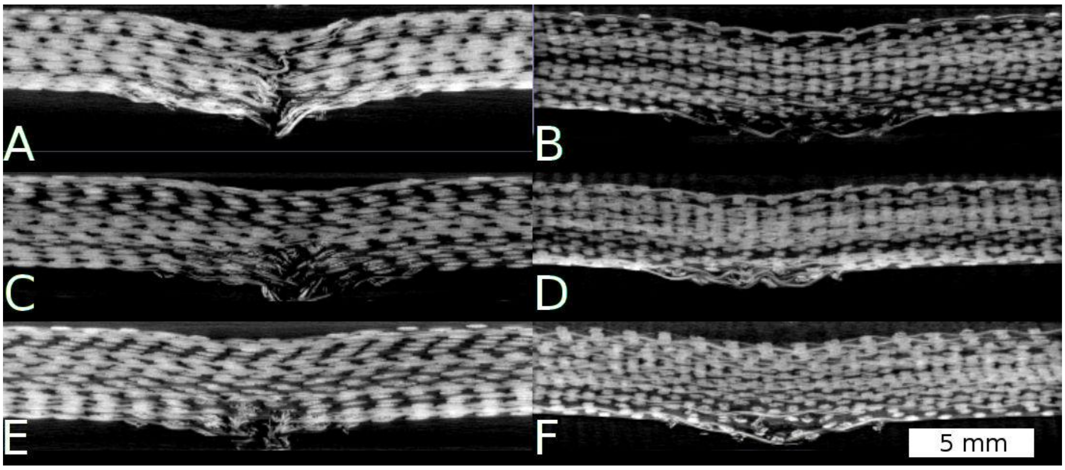

Figure 3. Reconstructions of the damaged zone in samples impacted at 27 J: A) COMP, B) NEAT, C) HNC, D) HCN, E) HCNC, F) HX

The high values of recovered energy [13] coupled with the reduced fibre breakage indicated that the matrix layering of HCN and HCNC can withstand much higher impact loads before composite failure with respect to the conventional compatibilized laminate configuration (COMP). The HCN configuration revealed the best overall performance among all IGIS designs but its asymmetric configuration can be used only if a preferential static or impact load direction can be identified, otherwise, for symmetric loads, the HX design could be the most suitable layering.

4. Conclusions

Hybrid thermoplastic composites based on polypropylene and glass fibre woven fabrics were prepared according to a new hybridization design, based on the gradation of the interlaminar interface strength (IGIS) without affecting the reinforcement configuration. In not graded (conventional) composites, the presence of the coupling agent increases the capability to transfer loads from the matrix to the fibres thus improving the flexural properties at the expense of the low velocity impact resistance. A reduced interface strength, in not compatibilized composites, permits to dissipate a higher amount of energy through sliding mechanisms and friction between fibres and matrix.

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The IGIS design allowed to prepare composites with high flexural properties coupled with high impact resistance and, as also supported by low-velocity impact tests, can be an effective way to retain good mechanical properties while allowing significant increases in the impact damage tolerance. In fact, different IGIS configurations showed limited damaging of fibres after an impact capable of inducing severe fibre breakage and laminate failure.

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