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# Impact response of Kevlar composites with nanoclay enhanced epoxy matrix

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ABSTRACT

Kevlar fibres have been widely used as impact-resistant reinforcement in composite materials. However, there are very few works about the effects of nanoclays on the impact strength of Kevlar/epoxy laminates. Therefore, this paper intends to study the ideal amount of nanoclays to obtain the best impact performance. Nanoclays Cloisite 30B were dispersed in the epoxy system of at 1.5%, 3% and 6% in weight. For better dispersion and interface adhesion matrix/clay, nanoclays were previously subjected to a silane treatment appropriate to the epoxy resin. The laminates manufactured with epoxy resin filled by 6 wt.% of nanoclays shown the best performance in terms of elastic recuperation and penetration threshold. The opposite tendency was observed for the displacement at peak load. However marginal benefits can be found when compared the results obtained for laminates filled by 3% and 6% of nanoclays.



## 1. Introduction

The poor tolerance to accidental low velocity impacts of composite laminates is yet a limitation to their use in many industrial applications. Various types of damages can occur, which are very dangerous because they are not easily detected visually and they can affect significantly the residual properties and structural integrity of those materials [1–7].

On the other hand nanoparticle reinforced materials have been widely studied and applied in numerous engineering and commercial areas due to their unique surface effects, increased chemical activity and particular physical properties [8,9]. Many researchers have reported great performance enhancements such as mechanical (strength and stiffness) and thermal properties were achieved by adding low concentrations of nanoparticles into polymers without compromising density, toughness or the manufacturing process [10–12]. Clay reinforcements, for example, have been shown to be effective reinforcements in neat polymeric structures [13–20].

Nanoclays used in fibre-reinforced polymeric composites in very low concentrations can improve the mechanical properties of these materials. Ferreira et al. [21] characterized the fatigue strength of a Kevlar/epoxy laminate composite as well as the benefits obtained by using a nanoclay-filled epoxy matrix. Filled composites exhibited tensile fatigue strengths 12% higher than unfilled matrices, but in three point bending the fatigue strength of filled composites was lower [21]. Studies developed by Timmerman et al. [22] reported that the transverse cracking in symmetric carbon fibre/epoxy laminates was significantly reduced when nanoparticle fillers were used. Siddiqui et al. [23] found that the initiation and propagation values of Mode I interlaminar fracture toughness of CFRP composites increased with increasing clay concentration. In particular, the propagation fracture toughness almost doubled with 7 wt.% clay addition. Low velocity impact response of carbon/nanoclay–epoxy composites was investigated by Hosur et al. [24]. Results of this study indicated that the infusion of nanoclay in the system reduced the impact damage though the impact response in terms of the peak load remained mostly unaltered. Reduced damage zone size was attributed to increased stiffness and resistance to damage progression of the nanophased laminates. Significant improvement in impact damage resistance and damage tolerance in the form of smaller damage area, higher residual strength and higher threshold energy level was obtained in CFRPs containing nanoclays by Iqbal et al. [25]. They reported that 3 wt.% clay was an optimal content for the highest damage resistance. On the other hand, the nanoclay presence in the epoxy matrix induced the transition of failure mechanisms of CFRP laminates during the CAI test, from the brittle buckling mode to more ductile, multilayer delamination mode. Studies performed by Ávila et al. [26] showed that the nanoclay presence in fibre glass/epoxy composites led to a more intense formation of delaminated areas after a low-velocity impact test. This phenomenon can be attributed to interlaminar shear forces caused by the intercalated nano-structures

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inside the epoxy system. However, the energy absorption of these laminates increased by 48% with dispersion of 5 wt.% of nanoclays. In terms of sandwich composites, Hosur et al. [27,28] showed that these materials with nanophased foam sustained higher loads and had lower damage areas compared to neat sandwiches. Sandwich composite plates made of fibre glass/nano-modified epoxy face sheets with different nanoclay contents, and polystyrene foams were prepared by Ávila et al. [29]. The results showed that the addition of 5 wt.% of nanoclay led to more efficient energy absorption and the failure modes were affected by the nanoclay contend. Other important aspect supported by Li et al. [30], for sandwich composites submitted to low velocity impact loads, is the occurrence of impact weave propagation between materials (face sheets and core). However, Ávila et al. [31] demonstrated that nano-modified laminated composites had their natural frequencies altered by nano-structures formed inside the laminate.

Aramid fibres are a very important reinforcement for military and civil systems. Their high degree of toughness, associated with the failure mechanism of aramids, and damage tolerance promotes good impact/ballistic performance. Several studies can be found in literature about impact response of composites reinforced by these fibres [32–38], however, there are very few works about the effects of nanoclay on the impact strength of Kevlar/epoxy laminates. Reis et al. [39] have performed the only study into this subject and compared two different fillers, cork powder and nanoclays Cloisite 30B. Nanoclays promoted higher maximum impact loads, lower displacements, the best performance in terms of elastic recuperation and the maximum residual tensile strength [39]. The filler content employed was 3 wt.% of the epoxy resin–hardener mixture [39] and the present work intends to find the ideal amount to obtain the best impact performance. The results of the present paper are discussed in terms of load–time, load–displacement, energy–time diagrams and damage. The impacted plates have been inspected by ultrasonic techniques and the residual strength was obtained by tensile tests. In fact, CAI (compression after impact) tests are suggested by the bibliography to evaluate the residual strength after impact in composite laminates, however, the aramid fibres fail by a series of small fibril failures and the amount of energy absorbed depends of the fibre strength [39].

## 2. Material and experimental procedure

Twelve ply laminates, all in the same direction, of woven bidirectional Kevlar 170-1000P (170 g/m$^2$), were prepared by hand lay-up and the overall dimensions of the plates were 330 × 330 × 3 (mm). SR 1500 epoxy resin and a SD 2503 hardener, supplied by Sicomin, were used. The system was placed inside a vacuum bag and a load of 2.5 kN was applied for 24 h in order to maintain a constant fibre volume fraction and uniform laminate thickness. During the first 10 h the bag remained attached to a vacuum pump to eliminate any air bubbles existing in the composite. The post-cure was followed according to the manufacturer‘s datasheet (epoxy resin) in an oven at 40 °C for 24 h.

Composite laminates with filled epoxy matrix by organoclays Cloisite 30B was produced by the same manufacturing process. In order to improve the dispersion and interface adhesion matrix/clay, the nanoclays were subjected to a special treatment appropriate to the epoxy resin. Surface treated clays were custom formulated by CTA Ltd. using chemical treatments using high shear mixing techniques, which is subject to patent applications. These may be defined as an organically modified layered silicate with a tetrahedral–octahedral–tetrahedral T–O–T basic structure with an additional surface layer treatment to give it enhanced dispersive characteristics in a resin compared to commercially available nanoclay (e.g. with granule size <50 μm, heavy metal content <100 ppm, and volatile content <1%).

The epoxy resin was heated in a glass beaker at 75 °C to decrease the viscosity and the nano-additives were added. Nanoclays were dispersed in the epoxy system of at 1.5%, 3% and 6% in weight. The mixture was conducted by using a high speed shear mixer at a shear rate of 2500 rpm for 1 h and followed by sonication (using an ultrasonicator) for 3 h to further disperse the clay. The resin temperature was maintained at 75 °C using a hot water bath during the mixing process. After sonication, the translucent colour of the epoxy/clay mixture indicated a uniform distribution of particles.

The samples used in the experiments were cut from these plates to square specimens with 100 mm side and 3 mm thickness (100 × 100 × 3 mm). Low-velocity impact tests were performed using a drop weight-testing machine IMATEK-IM10. More details of the impact machine can be found in [39]. An impactor diameter of 20 mm with a mass of 3.005 kg was used. The tests were performed on square section samples of dimensions 75 × 75 mm and the impactor stroke at the centre of the samples obtained by centrally supporting the 100 × 100 mm specimens. According with preliminary studies developed by the authors [39] the impact energies used in present work were 6, 12 and 21 J which correspond to an impact velocity of 2, 2.83 and 3.74 m s$^{-1}$, respectively. These energies were previously selected in order to enable the measuring of the damage area, but without promote perforation of the specimens [39]. For each condition, five specimens were tested at room temperature. After impact tests, the specimens were inspected by a C-Scan technique to evaluate the position and the size of the damage zone. Subsequently, the specimens were cut again to be submitted to tensile tests in order to obtain the residual strength. In this process it was used a circular diamond saw and a very careful procedure was applied in order to not increase the defects. The final dimensions of the specimens were 100 × 40 mm which corresponds to the area analysed by C-Scan. The tests were carried out in an electromechanical Instron Universal Testing machine (Model 4206) at a strain rate of 5 mm/s and at room temperature.

## 3. Results and discussion

Impact tests were carried out at different incident impact energy levels, namely, 6, 12 and 21 J. Fig. 1 shows the load and energy versus time response of control samples, 1.5%, 3%, and 6% nanoclay laminates, respectively for these three energies. These curves represent the typical behaviour for each laminate and are in good agreement with the bibliography [39–42].

The load–time curves exhibit oscillations that, according to Schoeppner and Abrate [43], result from the elastic wave and are created by sample vibrations. It depends on the stiffness and the mass of the specimen and impactor being excited by the rapid variation of the cinematic magnitudes at the collision moment [44]. These curves are characterized by an increase in the load up to a maximum value, $P_{max}$, followed by a drop after the peak load. In all tests the impactor deforms the specimens and always rebound, which means that the maximum impact energy was not high enough to produce full penetration. However, for control laminates (Kevlar/epoxy), when tested at 21 J, after $P_{max}$ the force decrease and remains constant while the time increases. This untypical curve, compared with the others, correlates with the appearance of major damage but still with a non-perforating impact event.

The average impact time was also measured and it varied in the range of 7.5–8.4 ms for 6 J, 7.2–8.3 ms for 12 J and 7.0–8.2 ms for 21 J. In all cases the maximum time of contact, between impactor and plates, occurred for control laminates and this value decreased with the percentage of nanoclays in the resin. For laminates manufactured with resin filled by 6% of nanoclays the average time was 7 ms, which represent 14.6% less than that, occurring for laminates with neat resin.

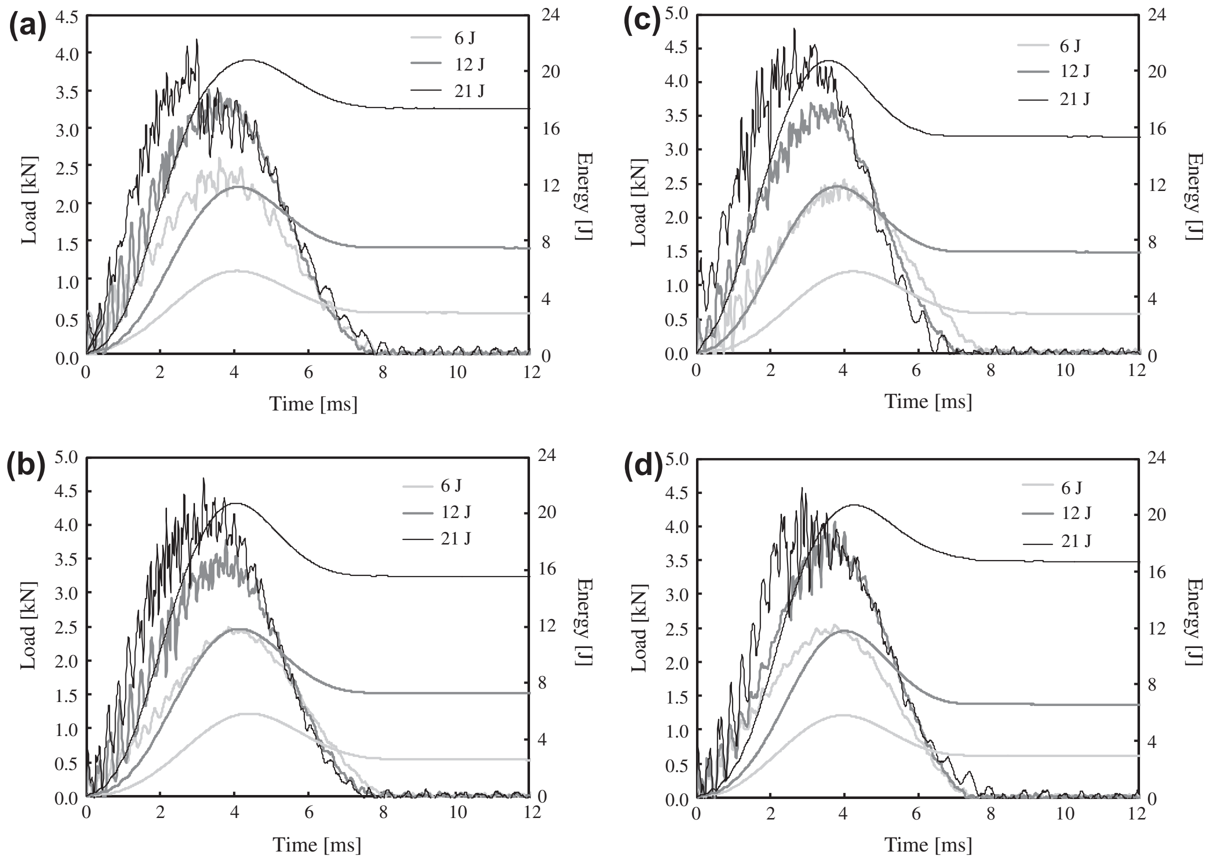


**Fig. 1.** Load and energy versus time response of: (a) control samples; (b) 1.5% nanoclay samples; (c) 3% nanoclay samples; (d) 6% nanoclay samples at three different energy levels.

From the curves that represent the evolution of the energy with time, it is possible to observe that the highest values of energy relate to smaller elastic recovery and, consequently, higher level of damage. The beginning of the plateau of the curve coincides with the loss of contact between the striker and the specimen, so, this energy coincides with that absorbed by the specimen [39,45].

Table 1 gives the average values of the peak load and the elastic recuperation for each laminate impacted at the three energy levels. The elastic energy was calculated as the difference between the absorbed energy and the energy at peak load from the curves in Fig. 1. The value of $P_{max}$ is very dependent on the impact energy. As expected there was an increase in the peak load as the energy levels were increased. A similar trend was also observed in the case of different weight percentages of nanoclay samples and agrees with studies reported by Hosur et al. [24]. For these authors, the maximum load increases almost linearly with increasing impact energy [24]. For the present study it is also possible to observe the same tendency when the resin is filled by nanoclays. The data can be fitted by a linear curve and the correlation coefficients obtained were 0.96 for neat laminates and 0.976–0.996 for composites loaded with nanoclays. Comparing the filler content effect, and for 21 J, the peak load increased around 4.8%, 19.2% and 23.5%, respectively, for 1.5%, 3% and 6 wt.% of nanoclays in comparison with control laminates. According to Gustin et al. [34] differences in maximum forces results in the different failure modes, because each sample type has different tensile and shear properties. According to authors, Kevlar laminates show tensile failure modes, demonstrated by bending at the striker perimeter and tearing at the centre of impact [34]. In terms of elastic recuperation the average values presented show that higher energies relate to lower elastic recuperation and consequently major damage. On the other hand, when the fillers are added better results can be found. The laminates manufactured with epoxy resin filled by 6 wt.% of nanoclays present best performance in terms of elastic recuperation.

**Table 1**
Average peak load and elastic recuperation.

| | Neat laminate | 1.5 (%) Nanoclay | 3 (%) Nanoclay | 6 (%) Nanoclay |
|---|---|---|---|---|
| *6 J* | | | | |
| Peak load (kN) | 2.52 | 2.59 | 2.63 | 2.71 |
| Elastic recuperation (%) | 49.55 | 51.91 | 53.59 | 55.03 |
| *12 J* | | | | |
| Peak load (kN) | 3.59 | 3.64 | 3.75 | 4.09 |
| Elastic recuperation (%) | 37.28 | 38.96 | 43.24 | 44.98 |
| *21 J* | | | | |
| Peak load (kN) | 4.17 | 4.37 | 4.97 | 5.15 |
| Elastic recuperation (%) | 14.96 | 21.41 | 24.65 | 26.94 |

However, for the impact energy of 21 J, if the value obtained relatively to the control samples is around 80.1% higher when compared with the samples filled by 3% of nanoclays the difference is only 9.3% higher. Therefore, it is possible to conclude that filler content higher than 3% of nanoclays promotes marginal improvements in terms of elastic recuperation.

Aktas et al. [46] used an energy profile diagram (EPD) to represent the relationship $E_i$ versus $E_a$ (where $E_i$ is the impact energy and $E_a$ is the absorbed energy) and to characterize some impact properties such as pure elastic limit, penetration and perforation thresholds. According with these authors, the penetration threshold can be defined as the point where the absorbed energy equals the impact energy for the first time. Namely, at penetration threshold, the impactor sticks into specimens and does not rebound any more [46]. Fig. 2 shows the energy profile diagram (EPD) of the laminates considered in this study. It is possible to observe that all data points are close and below of the equal curve, therefore, the absorbed energy never equals the impact energy and, according to Aktas et al. [46], the penetration threshold was not reached. In this region, the excessive energy is consumed to rebound the impactor and the damage extent is dependent on the impact energy. For each laminate the data can be fitted by a polymonial curve, $\Delta E = \alpha \cdot E_0^2 + E_0\beta + \delta$, where $\Delta E$is the energy absorbed, $E_o$ is the impact energy and $\alpha, \beta, \delta$ are constants presented in Table 2. This equation is more reasonable than the linear relationship between impact energy and the energy absorbed presented by Shim et al. [47]. Finally, Fig. 3 shows data points of $E_e$–$E_i$ variation and corresponding polynomials. The roots of these equations give energy points where $E_i/E_a$ = 1, i.e. where $E_e$ = 0. Therefore, the higher roots represent the penetration thresholds and values of 30.9 J, 32.9 J, 43.5 J and 44 J were found for neat laminates and laminates with 1.5%, 3% and 6 wt.% of nanoclays, respectively. In fact the polynomial fit was done only with three values of impact energies, which can promote some imprecision in terms of penetration threshold. However, it is evident that the fillers increase the penetration threshold and, according to these results, the higher value was obtained with 6 wt.% of nanoclays and occurs at impact energy around 44 J. Relatively to the control samples this improvement is around 42.4% higher and, one more time, this value is only 1.1% higher when compared with the samples filled by 3% of nanoclays.

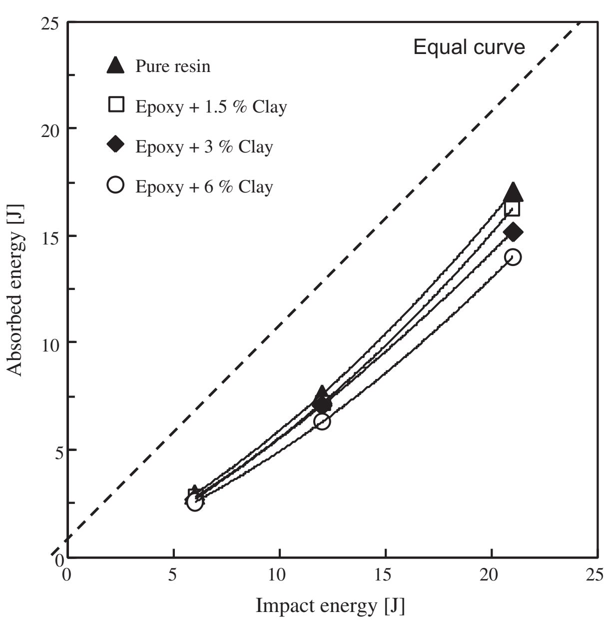


**Fig. 2.** Energy profile diagram of the laminates tested.

**Table 2**
Constants of the equation $\Delta E = \alpha \cdot E_0^2 + \beta E_0 + \varepsilon$ for different laminates.

| Laminates (%) | $\alpha$ | $\beta$ | $\varepsilon$ |
|---|---|---|---|
| Neat | 0.0184 | 0.4437 | −0.3747 |
| Epoxy + 1.5 clay | 0.0187 | 0.3927 | −0.2307 |
| Epoxy + 3 clay | 0.0113 | 0.5283 | −0.8767 |
| Epoxy + 6 clay | 0.0157 | 0.3397 | −0.032 |

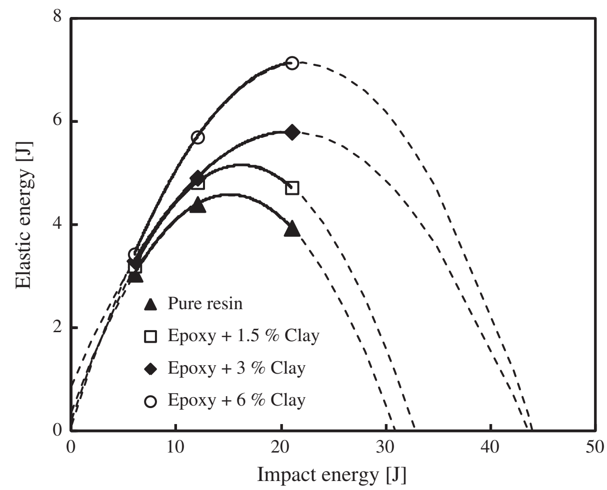


**Fig. 3.** Identification of penetration threshold.

On the other hand, according with Kang and Kim [32], for non-perforating impact test, at the point of maximum impactor displacement, the incident energy is fully transferred to the specimen. After the maximum displacement, the specimen transfers elastically stored impact energy back to the rebounding impactor. Therefore, the ratio of rebounded energy to stored energy could be a parameter of elastic/plastic properties. Table 3 presents the values obtained for all laminates. It is possible to observe that the elastic/plastic parameter increases with nanoclay content and decrease significantly when the energy increases (so, the damage increases).

It can be concluded that the laminates manufactured with epoxy resin filled by 6 wt.% of nanoclays presents the best performance in terms of elastic recuperation and penetration threshold compared to the neat laminates. This can be explained by the different damage mechanisms that occur in the laminates. In fact when the impactor strikes the specimen surface, the given impact energy ($E_i$) by the impactor can be classified into two quantities: elastic energy ($E_e$), which is stored elastically in the specimen and transferred back to the impactor, and the absorbed energy ($E_a$) [32]. For Kang and Kim [32] the absorbed energy can be divided into two categories; one is absorbed by the specimen ($E_{ab}$) while the other is absorbed by the testing system ($E_{sys}$). The total energy absorbed by the specimen has three components: membrane energy ($E_{mb}$), bending energy ($E_{bd}$) and energy absorbed by damage creation ($E_{del}$). Of course $E_{del}$ includes in delamination, fibre breakage, matrix cracking, etc. Kang and Kim [32] found, for

**Table 3**
Elastic/plastic properties of the laminates.

| Energy | Neat laminate | 1.5 (%) Nanoclay | 3 (%) Nanoclay | 6 (%) Nanoclay |
|---|---|---|---|---|
| 6 J | 1.03 | 1.14 | 1.22 | 1.33 |
| 12 J | 0.58 | 0.67 | 0.69 | 0.90 |
| 21 J | 0.23 | 0.29 | 0.38 | 0.49 |

Kevlar woven laminates, that the delamination was initiated at the centre of impact, and propagated to the directions of the warp and filled fibres. Due to the existence of warp and fill reinforcements the impact damages initiated by matrix cracks and were propagated along their length. However, energy absorbed by other mechanisms remains small compared to the energy absorbed by the major energy absorption mechanisms such as delamination and shape deformation.

All samples were inspected by C-Scan technique in a square area of 40 $\times$ 40 mm, containing the impact zone. Fig. 4 shows the typical images obtained for the laminates tested at 21 J, however, they are representative of the other ones. The inspection was made on the opposite side and, in this context, the blue colour represents the main damage promoted by the impact loads. It is possible to observe that the clays increase de damaged area, relatively to the control samples, for all energies used. The damaged area (blue area) was measured by Image-Pro Plus software and it was analysed in terms of the quotient between the blue area and the square area inspected (1600 $mm^2$). Relatively to the control samples, the damaged area increases, in terms of average values, 52.8%, 228.3% and 310.2% for the laminates filled by 1.5%, 3%; and 6% respectively. When compared the laminates with 3% and 6% of nanoclays the difference is around 24.9% higher for the last ones. In previous studies Reis et al. [39] studied the damage mechanisms for Kevlar composites with pure epoxy resin and with resin filled by 3% of nanoclays. For control laminates it was notorious that occurs a severe damage in the region of the impact point and it is characterized by big deformation in the thickness direction. For laminates with resin filled by clays this behaviour is not observed and the brittle aspect of the resin was visible. The matrix filled by clays present higher stiffness and, consequently, its ductile behaviour decreases [21]. Fig. 5 shows some pictures of the front and back face of the damaged laminates with pure resin (control laminates) and with epoxy resin filled by 1.5% and 6% of nanoclays. It is evident that the clays decrease the deformation in the thickness direction and increase the internal damages, as shown in Fig. 4. Therefore, the damage mechanisms are different and can explain the better impact strength of the hybrid laminates with nanoclays.

In fact some system must absorb the energy of the projectile, but cannot be allowed to deform so extensively that the wearer of the armour is crushed in the process. Diagrams load versus displacement are presented in Fig. 6 showing the effect of nanoclays contend. The average values represented for energy of 12 J shows that the largest displacements occur with laminates manufactured exclusively with pure epoxy resin. These curves are representative of all energies and Table 4 presents all results obtained. When nanoclays are added to the pure resin the displacements decrease

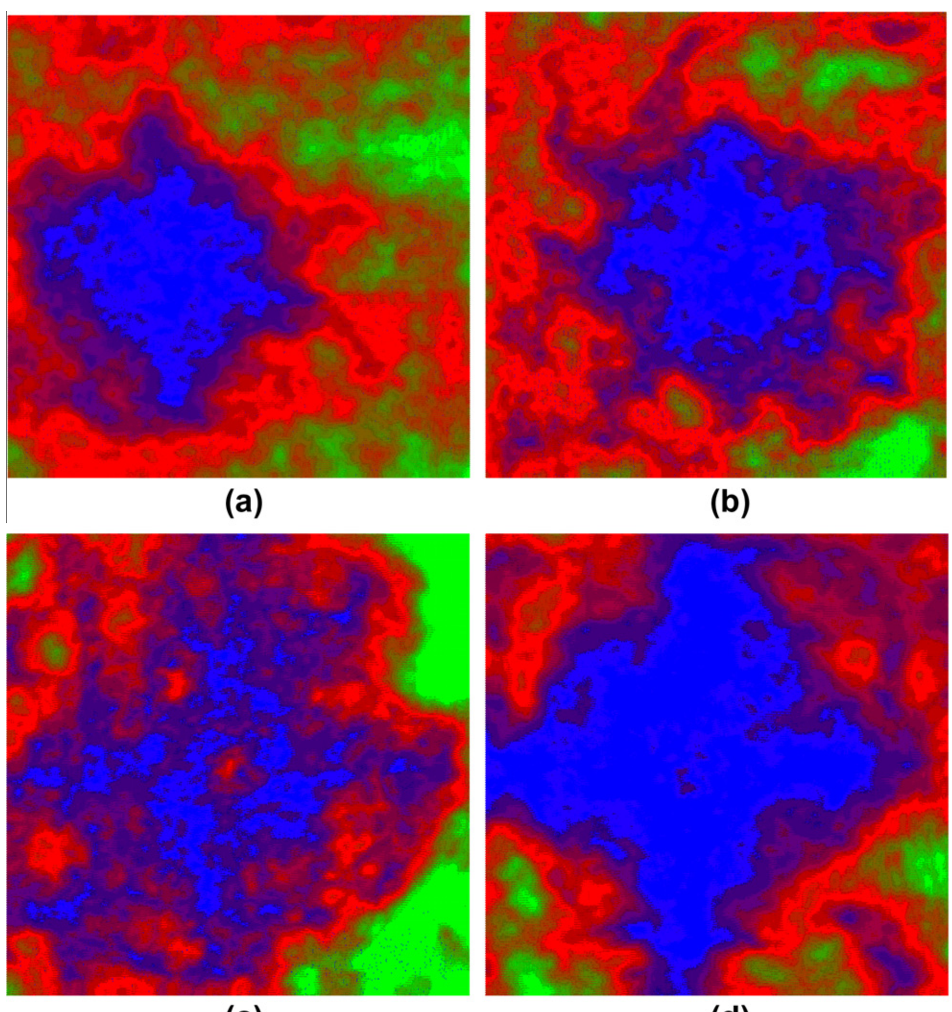


**Fig. 4.** C-Scan images of the damages for laminates, tested at 21 J, with: (a) epoxy resin; (b) 1.5% nanoclay samples; (c) 3% nanoclay samples; (d) 6% nanoclay samples.

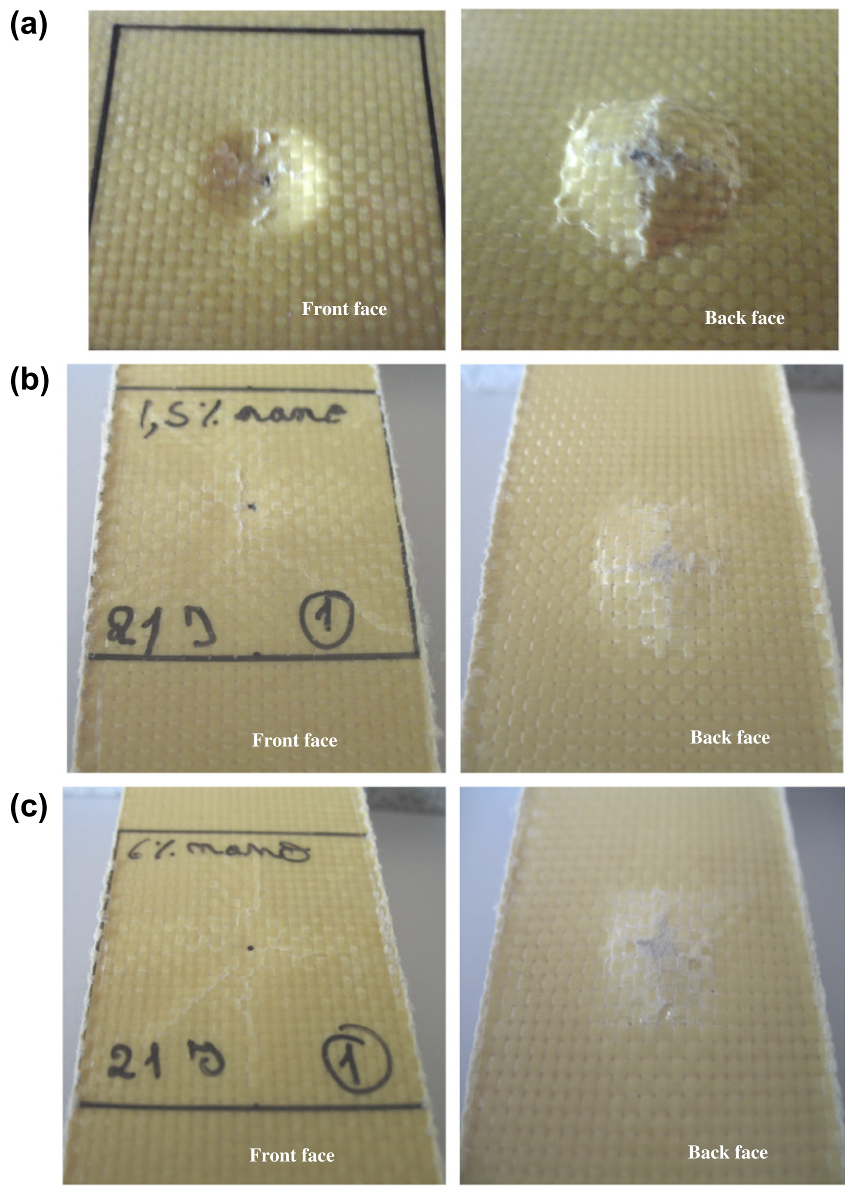


**Fig. 5.** Pictures of the damaged laminates, tested at 21 J, with: (a) epoxy resin [39]; (b) epoxy filled by 1.5% nanoclays; (c) epoxy filled by 6% nanoclays.

and the best performance was achieved for laminates filled by 6% of clays. In this case, for 21 J, a decrease around 12% was found relatively to the control samples. The same parameter compared with the value obtained for samples filled by 3% of nanoclays is only 0.6% lower.

After impact, residual tensile strength was obtained for each energy level as shows Fig. 7. It is evident that the increasing of the impact energy decreases the residual strength for all laminates and is consequence of the damage occurred. The control laminates present the lower residual tensile strength, for all impact energies, and the higher values are obtained with epoxy filled by 6% of nanoclays. For the energy of 6 J the residual strength is very close, because the damage effect is very similar independently of the laminates. After this value the fillers content presents a significant influence in residual strength of the laminates. For example, the laminates filled by 6% of nanoclays after impacted with energy of 21 J present residual strengths 43.2% higher than occurred for control laminates. However, the value obtained relatively to the samples filled by 3% of nanoclays is only 4.4% higher.

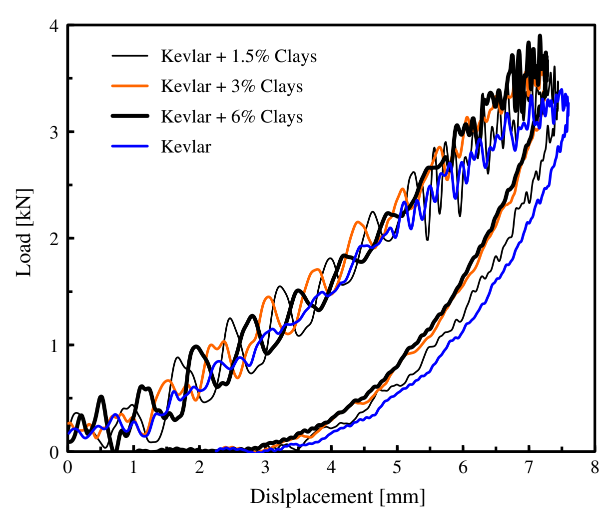


**Fig. 6.** Diagram load versus displacement for different materials tested at 12 J.

**Table 4**
Average displacement of the laminates.

| Energy | Neat laminate | 1.5 (%) Nanoclay | 3 (%) Nanoclay | 6 (%) Nanoclay |
|---|---|---|---|---|
| 6 J | 5.89 | 5.63 | 5.55 | 5.39 |
| 12 J | 7.60 | 7.31 | 7.27 | 7.09 |
| 21 J | 10.22 | 9.31 | 9.04 | 8.99 |

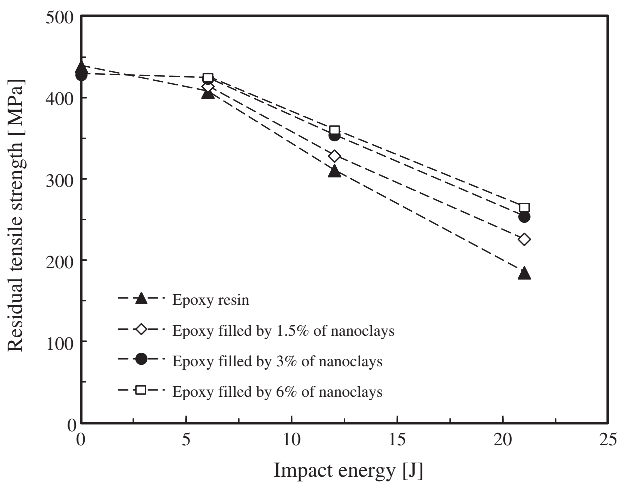


**Fig. 7.** Residual tensile strength versus impact energy.

## 4. Conclusions

The paper studied the low velocity impact response of a Kevlar/epoxy composite filled until 6% in weight by nanoclays Cloisite 30B, especially modified. The fillers adding increase the maximum impact load and the damaged area relatively to the control samples. The laminates manufactured with epoxy resin filled by 6 wt.% of nanoclays shown the best performance in terms of elastic recuperation and penetration threshold compared to the neat laminates. The opposite tendency was observed for the displacement at peak load, where the lower values were found for nanoclays filled composites. However marginal benefits can be found when compared the results obtained for laminates filled by 3% and 6% of nanoclays relatively to the other ones obtained between laminates filled by 1.5% and 3%. In terms of penetration threshold, for example, benefits around 1.15% and 32.22% were found, respectively.

The best performance in term of impact response obtained for the filled composites was also confirmed by the tensile residual strength which increases with filler content and the differences increase with the impact energy.

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