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ADVANCED EPOXY COMPOSITES OF IMPROVED IMPACT TOLERANCE

The materials aspects of improved low velocity impact tolerance have been reviewed. These include: tougher matrix systems, controlling fibre/matrix interfacial bond strength, using woven and mixed woven fibre fabrics, protective layers, stitching, Z-pinning, hybrid reinforcement, 3D structures, non-crimp fabrics NCF. For current structures, Z pinning shows the most promise, although the cost associated with this concept is currently high. For future structures, 3D woven materials show significant promise for impact tolerant design.

As an illustration to the problem selected experimental results from the authors' work on low velocity impact (rebound) behaviour were presented for hybrid glass/carbon/epoxy and glass/epoxy toughened with phenolic microspheres as well as aramid-glass with three different matrix types. The results showed that toughening epoxy matrix with phenolic microspheres had positive effect on absorbed impact energy (Figs 3, 4). The second phase acts to absorb significant amount of energy during fracture through mechanisms of crack front bowing and crack splitting. Changing to a tougher resin system has a number of advantages, most notably an increase in delamination resistance. However, other mechanical properties of tougher materials tend to be reduced, particularly compression dominated properties, which may suffer due to increased matrix compliance. Protective layer of aramid-glass fabric had minor effect on impact characteristics but was found promi-sing in terms of perforation resistance.

Interlayer woven carbon/glass-fibre/epoxy composites are used in practice in the construction of composite marine crafts at critical points of the glass/epoxy structure where improved stiffness is needed. The results obtained in this study for glass/carbon composites and in the previous work show (Figs 3, 4) that interlayer woven carbon/glass-fibre/epoxy composites exhibit high impact energy absorption combined with high strength. Impact damage resistance and impact damage tolerance of glass/carbon/epoxy composites depends on the stacking sequence of the laminate.

Key words: **impact behaviour, polymer composites, glass/carbon/epoxy laminates**

EPOKSYDOWE KOMPOZYTY WARSTWOWE O ZWIĘKSZONEJ TOLERANCJI UDARU

Przedstawiono przegląd sposobów poprawy tolerancji udarów w nowoczesnych laminatach polimerowych. Są to: wytrzymalsze i bardziej ciągliwe materiały osnowy, dobieranie odpowiedniego powiązania osnowy i włókien, stosowanie włókien w postaci tkanin, zszywanie preform z wielu warstw tkanin, łączenie włókien ciągłych w preformy za pomocą kompozytowych szpilek, stosowanie tkanin NCF z włókien ciągłych łączonych na grubości dodatkowym oplotem, wzmocnienie hybrydowe, warstwy ochronne z innych włókien. Jako ilustrację problemu przedstawiono niepublikowane, wybrane wyniki badań autorów naświetlające zachowanie kompozytów hybrydowych szklano-węglowych i szklanych o osnowie wzmocnionej mikrosferami z żywicy fenolowej oraz kompozytu szklanego z ochronną warstwą tkaniny aramidowo-szklanej. Wykazano, że wzmocnienie osnowy epoksydowej mikrobalonami fenolowymi ma pozytywny wpływ na absorpcję energii sprężystej (rys. rys. 3 i 4) dzięki mechanizmowi rozszczepiania końcówki pęknięcia i wyginania frontu pęknięcia. Wzmocnienie osnowy polimerowej utrudnia powstawanie delaminacji, jednak inne właściwości mechaniczne kompozytów o wzmocnionej osnowie ulegają nieznacznemu pogorszeniu. Ochronna warstwa aramidowo-szklana nie wpłynęła znacząco na poprawę absorpcji energii laminatu szklanego. Hybrydowe kompozyty wzmocnione warstwami włókien szklanych i węglowych są stosowane w praktyce w krytycznych miejscach konstrukcji kadłuba, gdzie wymagana jest podwyższona sztywność. Przedstawiono wyniki badań absorpcji energii laminatów o układzie warstw powierzchniowych: szkło/szkło/węgiel oraz węgiel/szkło i porównano z wcześniejszymi wynikami badań wpływu kolejności warstw węgiel/szkło na tolerancję udaru w próbie poudarowej wytrzymałości na ściskanie.

Słowa kluczowe: **odporność udarowa, kompozyty polimerowe, laminaty szklano-węglowo-epoksydowe**

INTRODUCTION

The frequent cause of in-service failure of laminates is low-velocity impact by dropped objects, resulting in extensive sub-surface damage (that may not be visible on the laminate surface) and in a considerable reduction in composite strength and stiffness [1]. Thus, it is appropriate to consider susceptibility to impact damage and impact tolerance of structural composite materials [2].

There is some confusion over the term impact tolerance with respect to PMC structures. There are two major issues, which may be addressed separately. The first - impact resistance deals with the ability to sustain

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a given impact threat with the minimum amount of damage. The second, impact damage tolerance, deals with the ability to sustain a given level of damage with minimum effect on structural performance. If a structure is impact resistant it does not follow that it will be impact damage tolerant: each of these aspects is controlled by different parameters. Therefore, the term impact tolerance deals with the overall ability to sustain a given impact with a minimum effect on the structure and thus combines both impact resistance and impact damage tolerance [3].

In order to enhance impact tolerance of composite laminates extensive researches have been performed which include: tougher matrix systems [4-6], interleaving methods [5, 7], controlling fibre/matrix interfacial bond strength [7, 8] using woven fibre fabrics [9, 10], hybrid reinforcement and protective layers [11-15], stitching [16-20], Z-pinning [3] non-crimp fabrics NCF [21].

In this study the materials aspects of improved impact tolerance have been reviewed. As an illustration to the problem authors' selected experimental results of low velocity impact behaviour of advanced laminates have been presented.

IMPACT TOLERANCE THROUGH NOVEL MATERIALS AND PROCESSING

There are two approaches to improve impact tolerance of fibre composites: one involves improvement of material properties of the composite constituents and the other - modification of fibre architecture. The former approach requires the improvements of fracture resistance of matrix material and the interface bond quality. The fibre-architecture modifications utilize textile structural preforms such as braiding, weaving, stitching and knitting to produce two-and three-dimensional (2D or 3D) fibre composites [10].

Tougher matrix systems

A number of techniques have been developed to improve toughness of resins used as matrices for impact tolerant composites. These include: the modification of epoxy resin with rubber such as carboxyl-terminated butadiene-acrylonitrile (CTBN) [22], addition of thermoplastic particles such as polyethersulphopne (PES) and polyetherimide (PEI) [23], the use of thermoplastic matrices such as PEEK [24, 25], the inclusion of thin, tough layers at ply interfaces [26]. The advantages of this concept are: similar costs and timescales, no need to redesign and usually the same processing route [27].

Woven laminates

During the impact event the weave acts to contain the shear and delamination cracks, significantly inhibiting the damage extent. In addition the compliant nature of woven laminates compared to unidirectional tape means that much of the energy is absorbed through structural response rather than damage formation. However, there are some disadvantages, poorer undamaged properties such as compression strength and stiffness. This is attributed to a high proportion of fibres not being aligned with the main loading direction. The reduction in stiffness is an important drawback since it will reduce the buckling performance. Compression strength is also reduced by the local crimping of the fibres (due to the weave) which promotes micro-buckling. These reduced undamaged properties lead to a need to redesign the structure which incur significantly increased costs and timescales [8, 28].

The poor undamaged performance of woven laminates can be improved by using a combination of wo-ven and unidirectional-tape-plies laminates. The woven layers impart improvements in the impact tolerance whilst the unidirectional layers improve the stiffness and undamaged strength [3].

Selective interlayers and hybrids

One alternative method for improving the impact tolerance is to insert layers of secondary material at critical locations in the stacking sequence. This can take the form of toughened particles on the ply surface, disc-rete toughened layers or as hybrid with glass or Kevlar plies at critical locations [15]. Selective hybrids have demonstarted improvements in both impact resistance and impact damage tolerance but the introduction of softened layers may reduce out-of-plane support on the load-bearing plies, promoting micro-buckling and compression failure. The need to increase thickness to achieve the same stiffness as the original component is also a problem, possibly requiring redesign [3].

Stitching

A technique which has been used to good effect is stitching in composites laminates [16-20]. The stitched material is usually Kevlar or glass fibres since these have the flexibility required for the high curvatures. Fabric preform is stitched through the thickness using Kevlar or glass fibres. Subsequently, the laminates are fabricated using VARIM process (vacuum assisted resin infusion molding). Under impact loading composites exhibit localized delamination (the spread of damage is cylindrical in contrast to the usual - conical). Stitching greatly improves impact damage tolerance of laminates manufactured from the tape. However, during the stitching of the preform the fibres are likely to get damaged and also are localised resin pools at the stitch location, which reduces undamaged properties.

Non-crimp fabrics (NCF)

There are problems with precision fibre placement of unidirectional prepregs as well as poor interlaminar strength and susceptibility to impact damage. In recognition of this, new class of textiles was developed - triaxial non-crimp fabric (NCF). This textile form (Fig. 1a) distinguishes itself from two-dimensional weaves and full three dimentional constructions by utilising a secondary yarn (polyester or aramid) that binds bundles of fibres together in differing orientations forming a blanket such that the reinforcing fibres are in near-zero-crimp state. The construction of a blanket of the triaxial non-crimp fabric consists of individual „plies" knitted together by a polyester or aramid yarn such that it has equal quantities of fibre in the 45 and - 45 directions and with the 0° content in the fabric equal to approximately 46% of the total weight [21]. The processing route of these materials is different to conventional autoclave route, using infusion or resin transfer instead. NCF's promise both reduced material cost and impro-ved damage tolerance, however the change in the fabrication route makes cost of certification as a replacement for conventional materials expensive and time consu-ming.

a)

b)

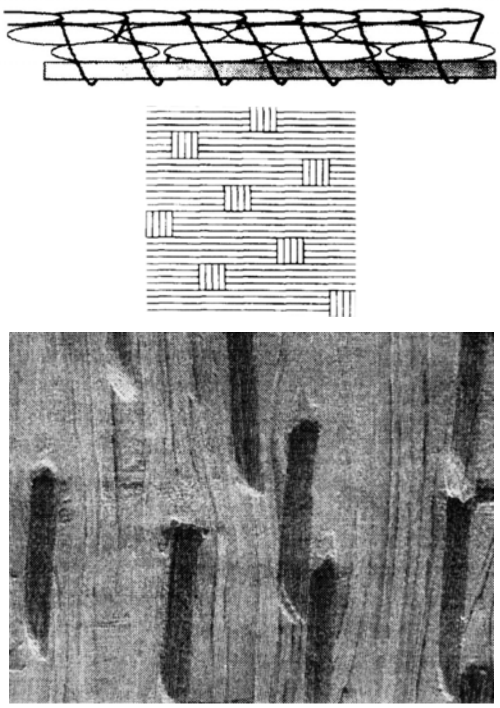

Fig. 1. Schematic illustration of non-crimp NCF fabric (a) [21] and electron micrograph of Z-pins on the fracture surface (b) [3]

Rys. 1. Schemat tkaniny NCF (a) [21] oraz obraz SEM szpilek (Z-pins) na powierzchni przełomu (b) [3]

Z-pinning

An alternative process to stitching is Z-pinning where reinforcing pins are inserted using an ultrasonic gun, which drives the pins into the laminate. Small diameter carbon fibre composite pins (less than 0.5 mm in diameter with a real density of typically 2%) are introduced into the laminate prior to cure (Fig. 1b). Z pins impart massive improvements in mode I and II toughnesses, impact resistance and damage tolerance, principally inhibiting delamintion growth. The loading mechanisms around the pins are complicated, processes such as deformation, splitting and fracture of the pins, pin pullout and ploughing all add to the work of fracture [3]. For current structures, Z-pinning shows the most promise, although the cost associated with this concept is currently high.

Three dimensional (3D) architecture

Composites reinforced with 3D fibre preforms exibit many ameliorating features, such as: enhanced strength and stiffness in the thickness direction, virtual elimination of interlaminar surfaces through the fully integrated fibre arrangement (making them free od delamination) and feasability of near-net-shape design and manufacturing [30]. However, the improvements in impact damage tolerance lead to reduction in the undamaged performance. The through-thickness fibres can also act as initiation sites for cracking and thus the threshold level at which damage starts to develop is not improved. Also it is more difficult to infuse resin into 3D composites, which can lead to defect formation in large components. More experimental work needs to be conducted on these materials, in particular development and modifica-tion of the standard test methods to allow their full characterisation. For future structures, 3D woven materials show significant promise for impact tolerant design.

MATERIALS AND EXPERIMENTS

Laminates were fabricated from woven orthogonal balanced fibre fabrics: carbon (RC660T), E glass (STR 66-110), aramid-glass (REA 390S). Carbon and hybrid aramid-glass fibre fabric were supplied by SP Systems (UK) and glass fibre fabric - by Krosno, Poland. Standard pro-adhesive treatment of the fibres for use with epoxy resins was provided by the suppliers. Laminates consisted of 5 plies of fabric impregnated with epoxy resin by hand lay-up. The resin was Epidian 52 (Organica-Sarzyna), typical Diglycidyl Ether of Bisphenol - A (DGEBA) (modyfied with active diluent), cured with amine hardener ET (20 wt.%). The approximate fibre volume fraction was V_f = 45÷50%.

All laminates consisted of 5 plies, the thickness was t = 2.2÷4.2 mm. The following reinforcement architecture has been used: 1/glass/carbon C: E/C/E/C/E, 2/ E/E/C/E/C, 3/glass/epoxy filled with phenolic microspheres (glass/epoxy +M), 4/REA/E/E/E/E/epoxy E52 - aramid-glass fabric REA (first layer from impact side) + 4 layers of glass fibre.

The rebound impact tests were conducted on the 100 mm square specimens in standard instrumented dropping weight tower CEAST DARTESTER at two energy levels: 2.7 and 9 J. Three specimens were tested for each material and impact energy. The load, displacement vs. time traces have been compared for all laminates. The absorbed (damaging) energy, and elastic rebound energy were found from load - displacement traces as shown in Figure 2.

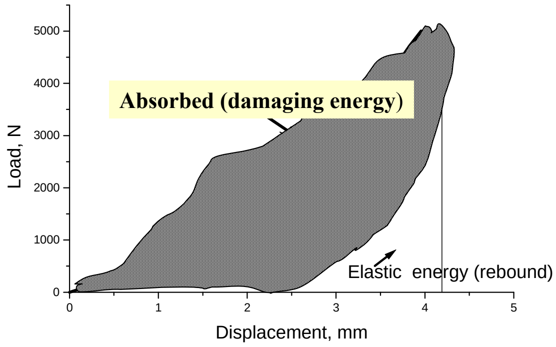

Fig. 2. Load-displacement trace in rebound impact test

Rys. 2. Wykres obciążenie-przemieszczenie w próbie udarowej z odbiciem próbki

RESULTS

Figure 3 shows load-time traces for all composites. Among hybrid laminates glass/carbon (60% glass) are clearly the stiffest and exhibit the highest P_m - maximum impact load. Carbon fibre laminate dominates, however, its thickness was 50% higher than the other laminates.

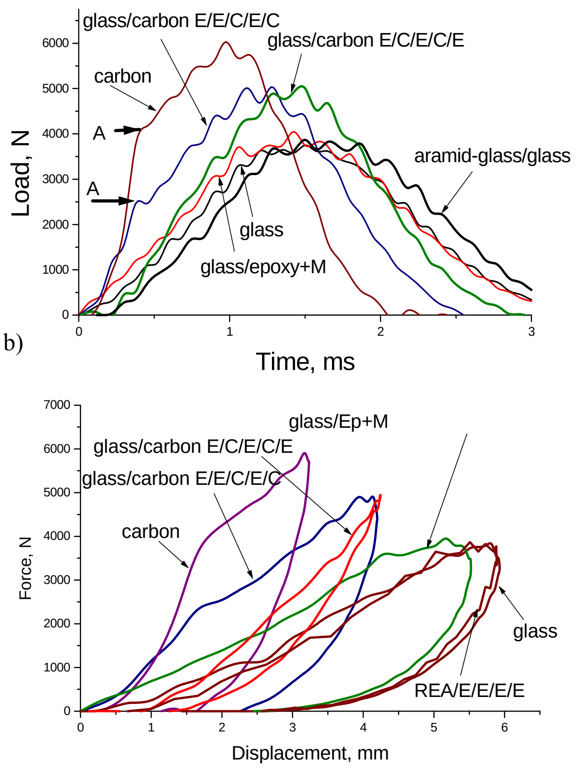

Fig. 3. Impact characteristics of different laminates: a) load-time, b) load-displacement traces. Points indicated by arrow A correspond to the first carbon ply failure

Rys. 3. Charakterystyki udarowe dla różnych laminatów: a) obciążenie-czas, b) obciążenie-przemieszczenie. Punkty wskazane przez strzałkę A odpowiadają pierwszemu pęknięciu warstw włókien

The behaviour of two glass/carbon composites is similar in terms of maximum load, however, stiffness represented by the slope of the initial load-time trace is slightly higher for unsymmentric composite: E/E/C/E/C (with carbon ply at the back) approaching the stiffness of pure carbon-fibre/epoxy laminate. Evidently, the stiffness of this ply controls the behaviour of the laminate until this ply fractures (arrow A). At this point the decline of the load-time curve changes and becomes similar to that exibited by glass fibre laminate, indicating that glass fibre plies took over the load carrying capacity of the laminate. The SEM image of impact-fractured glass plies are shown in Figure 5. Lower stiffness of symmetrical E/C/E/C/E laminate is related to the position of low modulus glass fibre ply - at the back of the laminate. However, carbon fibre ply constraints the deformation of glass fibre ply and the stiffness appears to be determined by the rule of mixtures. Energy absorbed was very different and uncomparable for both composites, because they were impacted from the opposite sides of the sample (resin-rich or fibre-rich), which, evidently, is of major importance. However, sample E/E/C and pure carbon/epoxy laminate were impacted in the same conditions and it can be seen that (Figs. 3b, 4) the hybrid composite absorbed much more energy.

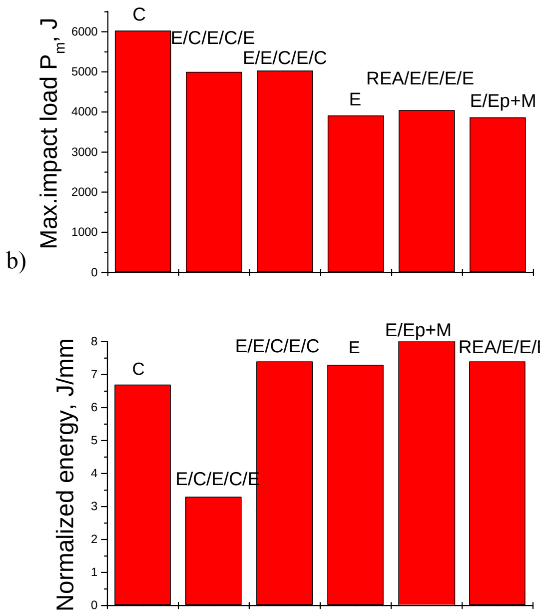

Fig. 4. Maximum impact load (a), absorbed energy E_a (b) for composites tested (impact energy 9 J)

Rys. 4. Maksymalne obciążenie (a), zaabsorbowana energia E_a (b) dla badanych kompozytów (b) (energia uderzenia 9 J)

For glass-fibre-based composites the maximum impact load was found very similar. However, it can be noted that glass/epoxy E52 modified with phenolic spheres exibits much higher impact energy absorption and that protective layer of aramid-glass fabric on the impact side had minor effect on impact characteristics.

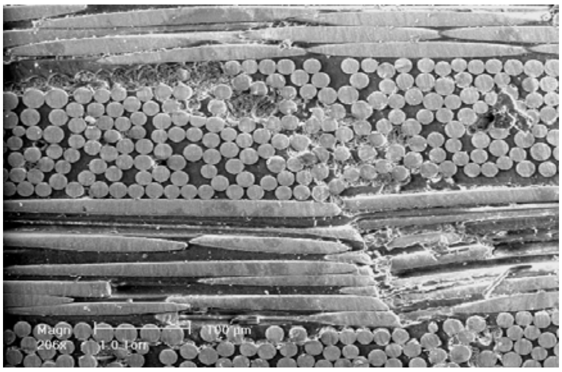

Fig. 5. SEM micrograph of the fractured glass-fibre ply in glass/carbon: E/E/C/E/C/epoxy composite. Impact energy 9 J

Rys. 5. Obraz SEM pęknięcia warstwy włókien szklanych w laminacie szklano-węglowym E/E/C/E/C o osnowie epoksydowej. Energia udaru 9 J

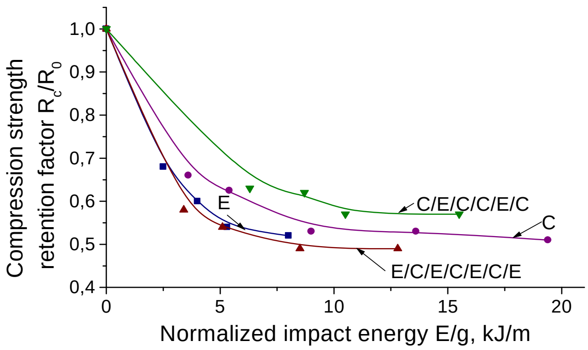

Fig. 6. Effect of stacking sequence on the compression strength retention factor in hybrid glass/carbon/epoxy composites

Rys. 6. Wpływ kolejności ułożenia warstw na wskaźnik degradacji wytrzymałości na ściskanie hybrydowych laminatów węglowo-szklanych o osnowie epoksydowej

DISCUSSION

The potential of graphite/epoxy composites is known to be reduced by the low impact strength which they characteristically exhibit. One method of alleviating this problem is to combine high static properties of graphite epoxy composite with a high impact strength of glass-fibre reinforced epoxy [31]. Carbon/glass/epoxy laminates are used in practice in the construction of composite marine crafts at critical regions of the glass/epoxy structure where improved stiffness is needed. The change in impact behaviour of this hybrid laminate, however, has to be considered. Although the impact performace of hybrid unidirectional glass/carbon/epoxy composites has been investigated in the past [31-34, 11] there is still the need to extend the data basis, particularly regarding impact tolerance of woven carbon/woven glass fibre fabric-reinforced laminates.

The results illustrated in Figures 3 and 4 show that interlayer woven carbon/glass fibre/epoxy composites exhibit high impact energy absorption combined with high strength which is the advantage of this material when compared with pure glass or carbon/epoxy laminates. The results obtained in this study and in the previous work [13, 14] on symetrical C/E or E/C laminates show that impact damage resistance and impact damage tolerance of glass/carbon/epoxy composites depends on the stacking sequence of the laminate. It was found [14] (Fig. 6) that the residual compressive strength retention factor is improved for the laminate with outer carbon layers C/E/C when compared with the E/C/E configuration. Further studies of impact tolerance and failure mechanisms of unsymmetric laminates are in progress.

Hybrid interlayer composites were examined by Jang et al. [10]. They found that the interlaminated hybrids generally showed slightly lower energy dissipation than that predicted by the rule of mixtures (taking into account) the properties of individual constituent lamina-tes. However, the maximum loads often showed positive synergism. In the present study this has been confirmed (in order to predict the respective properties by the rule of mixtures values from Figure 2 had to be divided by laminates' thicknesses). This can be accounted by the negative effect of thermal stresses resulting from dissimilar thermal expansions of constituent plies.

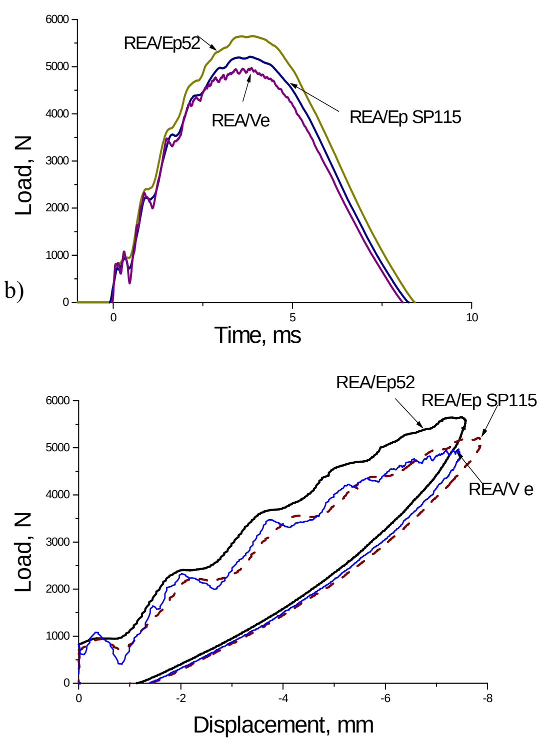

Fig. 7. Effect of matrix type (vinyl ester, modified epoxy resin E 52, epoxy SP115 (SP Systems)) on load-time (a), load-displacement traces (b) of hybrid aramid-glass-fibre reinforced composites

Rys. 7. Wpływ rodzaju osnowy polimerowej na przebiegi charakterystyk obciążenie-czas (a), obciążenie-przemieszczenie (b) dla kompozytów wzmocnionych hybrydową tkaniną aramidowo-szklaną

The results showed in this work highlight the importance of matrix properties. Toughening epoxy matrix with phenolic microspheres had positive effect on energy absorbed (Fig. 3) with only a slight decrease in flexural strength [35] and, interestingly, no observable loss in impact stiffness. The second phase acts to absorb a significant amount of energy during fracture through mechanism of crack front bowing, and crack end splitting [36]. Changing to a tougher resin system has a number of advantages, most notably an increase in delamination resistance. However, other mechanical properties of tougher materials tend to be reduced, paricularly compression dominated properties, which may suffer due to increased matrix compliance [3].

In order to illustrate the matrix effect on impact behaviour of hybrid laminates three different resins were examined by the authors: vinyl ester, epoxy modified E52% and epoxy SP115 (SP Systems) with aramid-glass-fibre fabric reinforcement REA. Only slight differences in the behaviour of the laminates were observed (Fig. 6) with some advantage of modified epoxy resin. This contrasts with the relative static flexural behaviour of these composites [35]. Vinyl ester matrix laminate REA/V shows 18% higher flexural strength than REA/E due to high adhesion efficiency of glass fibres and vinyl ester resin. However, on impact extensive aramid fibre/vinyl ester resin debonding is observed [37], which decreases the load transfer from the matrix to the fibres and leads to lower maximum load P_m in vinyl ester matrix composites. The adavantage of epoxy resin E52 in terms of impact energy absorption (Fig. 7b) can be explained by its ductility, one of the parameters controlling impact energy absorption.

Although the protective layer of aramid-glass fabric on the impact side did not show effective in terms of absorbed energy it has the advantage of higher perforation threshol energy. It was found to be improved by 20% when compared with pure glass/epoxy composite [35], which is of importance in marine crafts applications.

CONCLUSIONS

The materials aspects of improved low velocity impact tolerance have been reviewed. These include: tougher matrix systems, controlling fibre/matrix interfacial bond strength, using woven fibre fabrics, stitching, Z-pinning, hybrid reinforcement, non-crimp fabrics NCF and others. For current structures, Z-pinning shows the most promise, although the cost associated with this concept is currently high. For future structures, 3D wo-ven materials appear to be most promising for impact tolerant design.

As an illustration to the problem selected experimental results from the authors' work on low velocity impact (rebound) behaviour were presented for hybrid glass/carbon/epoxy and glass/epoxy toughened with phenolic microspheres, glass/epoxy laminate with protective layer of glass-aramid fabric as well as aramid-glass fabric with three different matrix types. The following conclusions were drawn:

- Toughening epoxy matrix with phenolic microsphe-res had positive effect on absorbed impact energy due to energy absorption mechanisms: crack front bowing and crack splitting.
- Interlayer, woven carbon/glass-fibre/epoxy composites exhibit high impact energy absorption combined with high strength. Impact damage resistance and impact damage tolerance of glass/carbon/epoxy composites depends on the stacking sequence of the laminate.
- Protective layer of aramid-glass fabric had minor effect on impact characteristics but was found promising in terms of perforation resistance.

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Recenzent
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