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Andreas Kessler & Andrzej Bledzki

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Influence of the fiber/matrix-interphase on the post-impact properties of glass/epoxy-laminates

ANDREAS KESSLER * and ANDRZEJ BLEDZKI

Institut für Werkstofftechnik, Kunststoff- und Recyclingtechnik, University of Kassel, Moenchebergstraße 3, D-34109 Kassel, Germany

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Abstract—In this paper the post-impact cyclic-dynamic properties and the compression-after-impact strength of glass/epoxy laminates with different fiber surface treatment are discussed.

It was found that the fiber/matrix adhesion quality significantly affects the compression strength of glass/epoxy cross-ply laminates. The residual compression strength is a function of the adhesion quality at the fiber/matrix interface. The cyclic-dynamic tests have shown that the dynamic strength under tensional loading is qualitatively comparable to the results of the compression-after-impact test with respect to the examined materials. The dynamic stiffness of the plates is significantly reduced in the case of poor fiber/matrix adhesion compared to the specimen with good and medium adhesion. The damage growth in the impacted plates during the cyclic-dynamic testing is also a function of the adhesion quality. The stiffness decreases and the energy absorption increases much faster in the case of poor fiber/matrix adhesion which indicates the poor damage tolerance of these laminates. A linear relation was found between the accumulated dissipated energy and the specimen temperature rise.

Keywords: Compression-after-impact; cyclic-dynamic loading; fiber/matrix-interface; glass fiber treatment.

1. INTRODUCTION

Though it is well-known that the bond strength between fiber and matrix significantly influences the mechanical properties of composites, only few publications can be found in the literature where the influence of glass fiber treatment on the impact behavior is discussed [1–5]. In general, depending on the type of reinforcing fibers, composites with poor fiber/matrix adhesion absorb more energy under impact loading because of extensive delamination and debonding processes [6–8]. Kessler and Bledzki [9, 10] have shown that the adhesion quality between glass fibers and matrix does significantly influence the impact damage resistance of cross-

*E-mail: kutech@hrz.uni-kassel.de

ply and unidirectional glass/epoxy laminates. Depending on the fiber treatment, up to five-fold increased impact damping and a significant change in damage area was observed by the authors.

To achieve comprehensive information about the impact behavior of different fiber treated reinforced polymers, the post-impact properties should be determined. In general, the compression-after-impact (CAI) test is used to study the residual strength of impacted laminates [11–14] because of its sensitivity to delaminations. Other testing methods which are frequently mentioned in the literature are the bending test [15] and the cyclic-dynamic load test [16, 17]. Under cyclic-dynamic loading not only the residual strength can be determined; other important parameters to describe the material behavior are the dynamic modulus and the dissipated energy [18].

The influence of the fiber/matrix interphase was generally studied on materials with undefined treatment and architecture. The residual compression and bending strength of composites with 'optimized' fiber/matrix adhesion is generally higher compared to composites with poor adhesion, which can be explained by the higher delamination resistance of these materials [19–22]. The influence of the silane type and silane concentration on the compression after impact strength of silane treated glass plain-woven fabrics in vinyl ester matrix was studied by Hirai *et al*. [23]. They measured only a slight increase of the residual strength with increasing silane concentration.

More information about the influence of the fiber surface treatment on the post-impact properties can be achieved by cyclic-dynamic load tests. Gassan [24] found that the dynamic modulus of natural fiber reinforced plastics is less affected by impact loading when the fiber matrix adhesion is improved by coupling agents.

There is a lack of investigations about the influence of fiber surface treatment on the dynamic post-impact properties of glass fiber reinforced plastics in the open literature. For that reason, in this paper the post-impact cyclic-dynamic properties of glass/epoxy laminates with different fiber surface treatment are discussed. To get a comprehensive overview about the material post-impact properties, the compression-after-impact strength was also determined.

2. EXPERIMENTAL

2.1. Materials

E-glass fibers (12 μm, 63 tex) with three different surface treatments were used in this work. The following treatments were applied to achieve composites with varying fiber/matrix adhesion:

EP: Epoxy dispersion on the base of bisphenol A as binder and a γ-aminopropyltriethoxy silane coupling agent (good fiber/matrix-adhesion).

PU: As EP, but the EP-binder was substituted by a polyurethane dispersion (medium adhesion).

PE: High molecular polyethylene dispersion (poor adhesion).

Two Ciba Geigy epoxy systems were used as matrix:

556/917: Araldit LY 556 with the hardener HY 917 and the catalyst DY 070.

556/208/917: To study the influence of the matrix ductility, an epoxy flexibilizer resin CY 208 was added to the system 556/917.

The tension strength and elongation at break of the matrix systems are summarized in Table 1.

Cross-ply laminates with a $(0°, 90°, 0°, 90°)_{sym}$ lay-up (thickness 2 mm) and a fiber content of $54 \pm 3\%$ by weight were prepared on a laboratory winding machine.

2.2. Post-impact properties

2.2.1. Compression-after-impact (CAI). The tests were carried out according to DIN 65561. To reduce the material expense, the original specimen dimensions of 150×100 mm^2 were scaled down to 60×40 mm^2. The impact tests were employed on a falling weight impact tower. The specimens were clamped between two plates with a rectangular test window of 52×32 mm^2 and subjected to transverse impact with a spherical impactor (16 mm diameter) and a kinetic energy of 7.5 J, 12 J and 16 J (impactor mass: 1.3 kg).

The compression fixture imposed clamped boundary conditions on the load ends and simply supported boundary conditions on the side edges to prevent out-of-plane buckling of the test plates. The compression loads were applied parallel to the fiber orientation of the outer plies of the laminates with a cross-head speed of 1 mm/min; 5–6 specimens of each material were tested.

2.2.2. Cyclic-dynamic loading. Specimens with the dimensions of 150×16 mm^2 including 43 mm for each glassfiber reinforced end tab were cut from the plates. The fibers of the outer plies were orientated in the longitudinal direction of the specimens. On the falling weight impact tester the samples were clamped between two circular rings with a testing window of 40 mm. Transverse impact was applied to the center of the specimens with energies of 7.5 J, 10 J and 12 J (impactor mass: 1.3 kg). To exclude effects of the sharp edges of the plate fixture, the plates were supported by rubber rings. Since these rubber rings dissipate an undefined amount of energy, the energy absorbed by the laminates could not be measured accurately; 5–6 specimens of each material were tested. Since the damage extension reaches

Table 1.
Tension test data of the matrix systems

Matrix system	Strength (MPa)	Elongation at break (%)
556/917	72.0 ± 8	3.3 ± 0.8
556/208/917	74.7 ± 2	8.1 ± 1.5

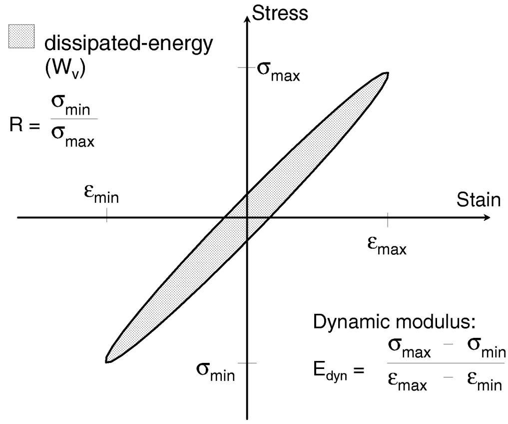

Figure 1. Hysteresis loop and definition of the dynamic modulus and the dissipated energy [25].

the plate edges during these tests, the damage area of the laminates should not be compared. For that reason the damaged area was not measured.

The undamaged and impact damaged specimens were subjected to increasing cyclic-dynamic tension loading on a 50 kN MTS testing machine (25 kN MTS load cell) with a stress ratio of $R = 0.1$. An MTS extensometer with an extended gage length of 50 mm and a travel of ± 1 mm was used to measure the strain. Starting with 80 MPa, the maximum stress was increased each 3000 load cycles by 20 MPa. The dissipated energy and the dynamic modulus of each load cycle were calculated according to Lazan [25] as shown in Fig. 1. The specimen temperature was measured by mounting a thermocouple on the damaged area of each specimen.

3. RESULTS

3.1. Compression-after-impact (CAI)

The variation of the compression strength of the tested laminates depending on the fiber surface treatment, the matrix and the impact energy is summarized in Fig. 2. It can be seen that the PE-fiber treatment leads to a reduced compression strength in the undamaged state (0 J). This indicates the poor fiber/matrix adhesion of these laminates. The flexibilizer slightly reduces the strength of the undamaged laminates with coupling agent (EP, PU). Since the tension strength of both matrix systems is about the same (Table 1), it seems that the fiber/matrix interphase is weaker in the case of the flexibilized matrix. Using PE-treated fibers, the fiber/matrix adhesion is extremly low and seems not to be influenced by the matrix system. Due to the poor bond strength, the residual strength of the ‘PE’-laminates reaches only about 100 MPa after an impact with 16 J. The laminates with good fiber/matrix adhesion show a significantly higher residual strength.

Since the damage extension reached the boundary of the impact plate fixture in some cases, the damaged area of the laminates cannot be compared. For that reason

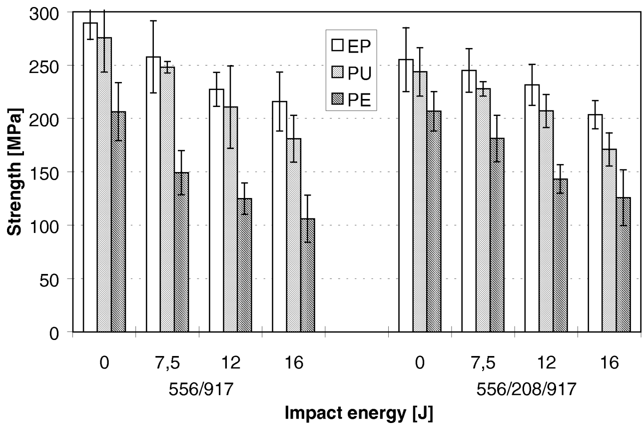

Figure 2. Compression strength and standard deviation of all laminates depending on the fiber surface treatment, the matrix and the impact energy.

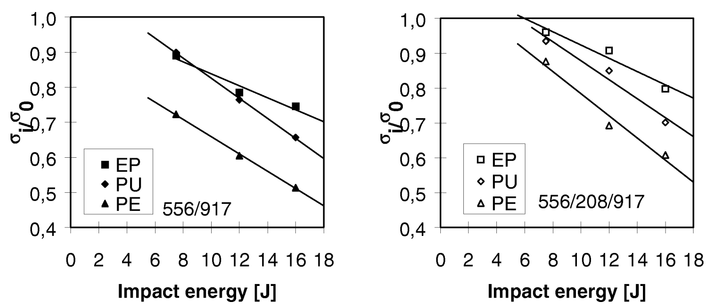

Figure 3. Relative residual compression strength depending on the fiber surface treatment, the matrix and the impact energy.

these values were not measured. But in general it can be said that, independent of the fiber type, the projected damaged area is smaller using the flexibilized matrix. In the case of the EP- and PU-treated fibers, the positive effect of the smaller damage of the laminates with the matrix 556/208/917 is superposed by the smaller strength of these composites. For that reason no significant influence of the matrix can be seen. In the case of the poor bond strength (PE), only the effect of the smaller damaged area for the plates with the flexibilized matrix took place, so that these laminates show a slightly higher residual strength compared to the 'PE'-plates with the matrix 556/917.

The relative strength of the damaged laminates is shown in Fig. 3. A higher level of relative strength can be observed in the case of the matrix 556/208/917, which means that for all types of fiber surface treatment, the flexibilizer CY 208 improves the damage resistance of the laminates. The highest damage resistance was determined on the plates with the EP-fiber treatment. At low impact energies the relative strength does not significantly differ between the laminates with

PU- and EP-fiber treatment. But at energies of about 16 J, the laminates with the strong fiber/matrix bond strength (EP) show the higher compression strength.

3.2. Cyclic-dynamic loading

The dynamic strength of the materials is shown in Fig. 4. Again, the strength of the undamaged laminates with the PE-treated fibers is lower compared to the other materials. Although an improvement of the dynamic stength can be expected for the laminates with the flexibilized matrix, no significant influence of the matrix was observed. Again it seems that the toughening effect of the flexibilizer is superposed by a slightly reduced bond strength between the flexibilized matrix and the coupling agent treated fibers.

The laminates with the PE-treated fibers show the lowest damage resistance. Due to impact, a strength reduction of up to 40% can be observed on the plates with PE-treated fibers. The EP-treatment leads to a significantly higher damage resistance (20% strength reduction). The laminates with the PU-treated fibers show a fair damage resistance compared to the other materials. Contrary to the CAI test, no significant influence of the matrix can be observed. This is due to the fact that, in tension loading, the strength is dominated by the amount of broken fibers, which is relatively insensitive to the matrix type.

Since the scatter of the dynamic strength is high, it is interesting to look also at other parameters. One is the dynamic modulus which is shown in Fig. 5 for two different materials. For the laminates with poor fiber/matrix adhesion (PE) an early stiffness reduction can be observed with increasing cycle number. This indicates the fast damage growth in the plates with PE-treated fibers and indicates the low damage tolerance of these materials. The lower stiffness of these plates in the beginning of the test is caused by the greater amount of damage due to impact loading. The reduction of the dynamic modulus due to impact is shown in Fig. 6.

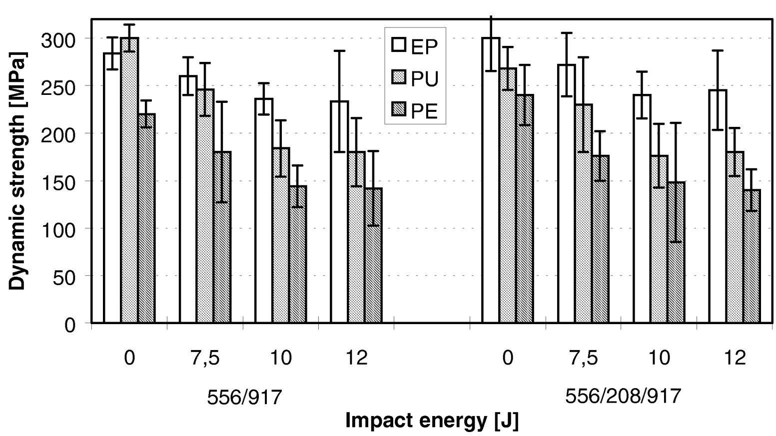

Figure 4. Cyclic-dynamic strength and standard deviation of all laminates depending on the fiber surface treatment, the matrix and the impact energy.

This characteristic value is calculated using the average modulus over the first 600 cycles of each test. Again, the PE-fiber treatment leads to the highest reduction of the dynamic modulus. The matrix system seems to be of minor importance.

Another important damage parameter is the dissipated energy and the accumulated dissipated energy (total dissipated energy) [18]. For two materials the accumulated dissipated energy depending on the impact energy is shown in Fig. 7. It

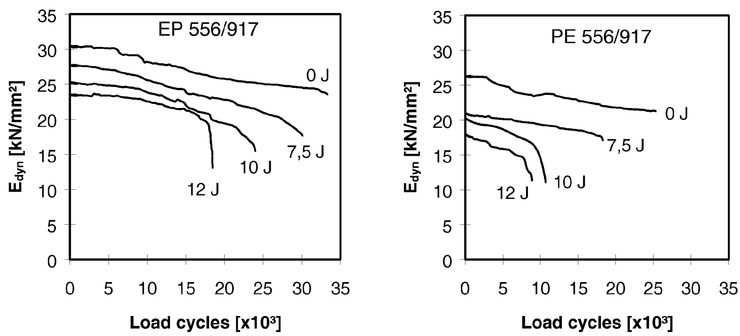

Figure 5. Dynamic modulus during cyclic-dynamic testing with increasing load of laminates with poor (PE) and good (EP) fiber/matrix adhesion in the matrix 556/917.

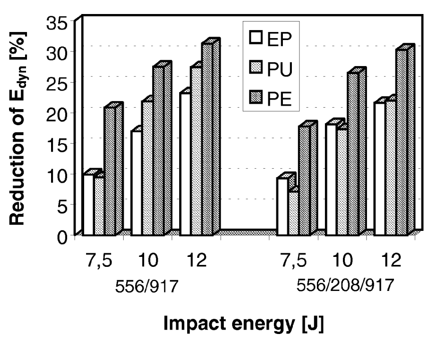

Figure 6. Reduction of the dynamic modulus caused by impact with different energies.

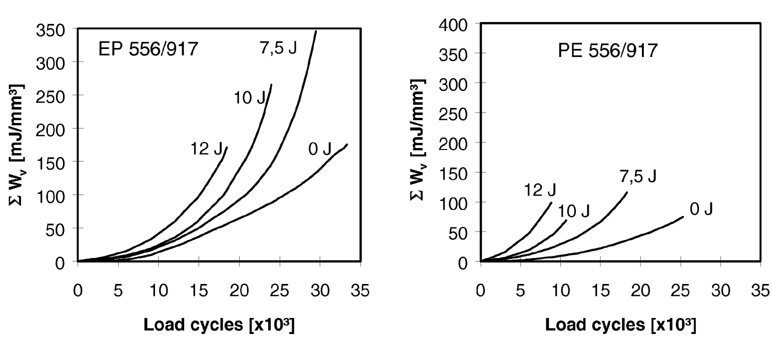

Figure 7. Accumulated dissipated energy during cyclic-dynamic testing with increasing load of laminates with poor (PE) and good (EP) fiber/matrix-adhesion in the matrix 556/917.

was found that the damaged laminates absorb much more energy during the early stage of the test than do the undamaged laminates (0 J). This is due to friction on crack surfaces and damage growth. In general, the impacted laminates with the PE-treated fibers absorb more energy until a given cycle number is reached than do the laminates with the EP-treated fibers. On the other hand, the laminates with poor fiber/matrix adhesion (PE) dissipate less energy until failure occurs.

An indirect measurement for the energy absorption of undamaged and damaged laminates is the temperature rise during the test. Figure 8 shows the temperature rise of the specimens from Fig. 7. A maximum temperature increase of 20°C can be observed, which is still significantly below the glass transition temperature of the resin. It can be seen that the temperature increases faster in the damaged laminates and in the laminates with the PE-treated fibers corresponding to the higher accumulated dissipated energy. If the accumulated dissipated energy is plotted against the temperature rise, a linear relation between the two values can be found (Fig. 9).

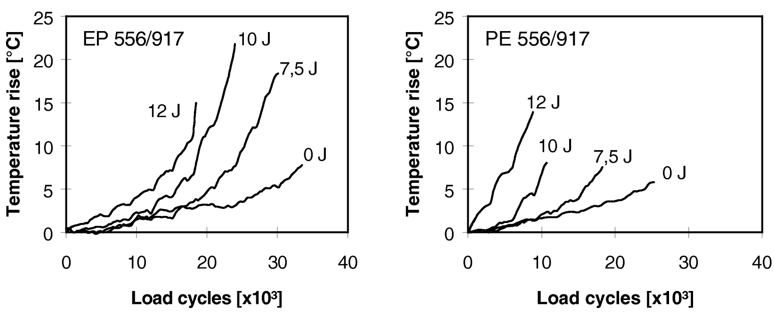

Figure 8. Temperature rise during cyclic-dynamic testing with increasing load of laminates with poor (PE) and good (EP) fiber/matrix adhesion (matrix 556/917).

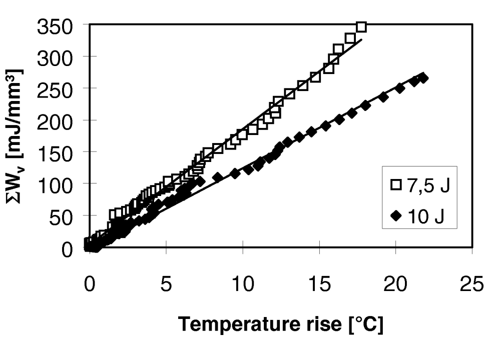

Figure 9. Correlation between the accumulated dissipated energy and the temperature rise of a specimen with EP-treated fibers and the matrix 556/917 after impact with 7.5 J and 10 J.

4. CONCLUSIONS

Laminates with defined architecture and defined different fiber surface treatment were investigated. The following conclusions can be drawn:

- The fiber/matrix adhesion quality significantly affects the compression strength of glass/epoxy cross-ply laminates. If these laminates are subjected to non-penetration impact, more extensive delamination can be observed in the specimens with poor adhesion. As a result, the residual compression strength is a function of the adhesion quality at the fiber/matrix-interface. By increasing the ductility of the matrix system, the post-impact strength can be improved. The highest compression-after-impact strength (damage resistance) was achieved for laminates with a strong fiber/matrix interface and a more ductile matrix system.
- The cyclic-dynamic tests have shown that the dynamic strength under tensional loading is qualitatively comparable to the results of the compression after impact test with respect to the examined materials. The advantage of cyclic-dynamic testing is that more information about the damage propagation can be obtained. It was found that the dynamic stiffness of the plates is significantly reduced in the case of poor fiber/matrix adhesion compared to the specimen with good and medium adhesion. The damage growth in the impacted plates during the cyclic-dynamic testing is also a function of the adhesion quality. The stiffness decreases and the energy absorption increases much faster in the case of poor fiber/matrix adhesion, which indicates the poor damage tolerance of these laminates. A linear relation was found between the accumulated dissipated energy and the specimen temperature rise.

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REFERENCES

1. G. Wacker, Experimentell gestützte Identifikation ausgewählter Eigenschaften glasfaserverstärkter Epoxidharze unter Berücksichtigung der Grenzschicht, PhD thesis, Institut für Werkstofftechnik, University of Kassel, Germany, Shaker-Verlag, Aachen (1996).
2. A. Kessler, A. K. Bledzki, G. Wacker and H. Frenzel, Einfluss der Grenzschicht auf die Daempfungseigenschaften von unidirektional verstaerktem Glas/Epoxidharz-Verbunden unter Impactbelastung, in: *Proceedings of the Conference Verbundwerkstoffe und Werkstoffverbunde*, Kaiserslautern, DGM Informationsgesellschaft mbH, pp. 395–400 (1997).
3. P. Yeung and L. J. Broutman, The effect of glass-resin interface strength on the impact strength of fiber reinforced plastics, *Polym. Eng. Sci.* **18** 62–72 (1978).
4. A. M. El-Habak, Effect of impact perforation load on GFRP composites, *Composites* **24** 341–345 (1993).
5. T. Morrii, H. Hamada, N. Ikuta, M. Desaeger and I. Verpoest, Effect of silane treated fiber on damage tolerance in glass fabric/vinylester laminates subjected to static and impact flexural

loading, in: *Proceedings of the Interfacial Phenomena in Composite Materials*, Eindhoven, pp. 131–132 (1995).
6. P. K. Mallick, in: *Fiber-Reinforced Composites*, L. L. Faulkner and S. B. Menkes (Eds), pp. 248–263. Marcel Dekker Inc. (1988).
7. A. A. J. M. Peijs, P. Catsman, L. E. Govaert and P. J. Lemstra, Hybrid composites based on polyethylene and carbon fibers. Part 2: Influence of composition and adhesion level of polyethylene fibers on mechanical properties, *Composites* **21** 513–521 (1990).
8. B. E. Anderson and X. N. Huy, Characterization of extended-chain polyethylene fiber reinforced polyethylene matrix composite, in: *Proceedings of the Materials Working For You in the 21st Century, 37th Int. SAMPE Symp. and Exhibition*, Vol. 37, pp. 850–863. Soc. for the Advancement of Mater. and Process Engng, Anaheim (1992).
9. A. Kessler and A. K. Bledzki, Low velocity impact behavior of glass/epoxy cross-ply laminates with different fiber treatments, *Polymer Compos.* **20** 269–278 (1999).
10. A. K. Bledzki, J. Gassan and A. Kessler, Loss-energy of composite materials — Part 2: Impact loading, *JTEVA* **27** 36–41 (1999).
11. M. De Freitas and L. Reis, Failure mechanisms on composite specimens subjected to compression after impact, *Comp. Struct.* **42** 365–373 (1998).
12. A. T. Nettles and A. J. Hodge, Compression-after-impact testing of thin composite materials, in: *23rd International SAMPE Technical Conference*, Covina, pp. 177–183 (1991).
13. J. C. Prichard and P. J. Hogg, The role of impact damage in post-impact compression testing, *Composites* **21** 503–511 (1990).
14. M. J. Pavier and M. P. Clarke, Experimental techniques for the investigation of the effects of impact damage on carbon fiber composites, *Compos. Sci. Technol.* **55** 157–169 (1995).
15. J.-K. Kim, D. B. Mackay and Y.-W. Mai, Drop-weight impact damage tolerance of CFRP with rubber-modified epoxy matrix, *Composites* **24** 485–493 (1993).
16. R. Jones, J. Paul, T. E. Tay and J. F. Williams, Assessment of the effect of impact damage in composites: Some problems and answers, *Comp. Struct.* **10** 51–73 (1988).
17. M. H. Beheshty and B. A. Harris, A constant-life model of fatigue behaviour for carbon-fiber composites: The effect of impact damage, *Compos. Sci. Technol.* **58** 9–18 (1998).
18. A. K. Bledzki and J. Gassan, Dissipated energy of composite materials — Part 1: Cyclic dynamic stress, *JTEVA* **26** 467–471 (1998).
19. W. J. Cantwell and J. Morton, The impact resistance of composite materials — a review, *Composites* **22** 347–362 (1991).
20. P. W. Manders and W. C. Harris, A parametric study of composite performance in compression-after-impact testing, *SAMPE J* **22** 47–51 (1986).
21. S. M. Lee, Influence of fiber/matrix interfacial adhesion on composite fracture behaviour, *Compos. Sci. Technol.* **43** 317–327 (1992).
22. M. Stuard and V. Altstädt, Carbon fiber surface treatment and effect on composite damage tolerance performance, in: *Proceedings of the 21st Int. SAMPE Techn. Conf.*, Anaheim, pp. 264–274 (1989).
23. Y. Hirai, H. Hamada and J.-K. Kim, Impact response of woven glass-fabric composites — 1. Effect of fiber surface treatment, *Compos. Sci. Technol.* **58** 91–104 (1998).
24. J. Gassan, Naturfaserverstärkte Kunststoffe-Korrelation zwischen Struktur und Eigenschaften der Fasern und deren Composites, PhD thesis, Institut für Werkstofftechnik, University of Kassel, Germany, Shaker-Verlag, Aachen (1997).
25. B. J. Lazan, *Damping of Materials and Members in Structural Mechanics*. Pergamon Press, Oxford (1968).