Original Paper

SiC- and carbon-fibre-reinforced glass under alternating bending stress loadings

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Dedicated to Prof. Dr. Erwin Roeder on the occasion of his 65th birthday

Alternating bending stress experiments are described which were performed with various fibre/DURAN-glass composites reinforced by Nicalon NL 202 SiC fibres and by two different types of carbon fibres, a high-tensile strength (ht) and a high-modulus (hm) fibre. Also the influence of unidirectionally homogeneous and inhomogeneous fibre distribution as well as the bidirectional 0°/90° ply distribution are studied. In contrast to previously investigated strain-controlled experiments the present stress-controlled experiments show after the pretreatment of 1000 alternating tensile-compressive stress cycles fatigue (beginning damage) already at amplitudes below the limit of pure elasticity (below bendover stress) of a simple bending-load experiment. Despite of this difference the "training effect" of the previous investigation, the increase of the bendover stress with increasing stress amplitude after 1000 alternating-load cycles, is found to be similar and can be established also for the present experiments. The carbon-fibre-reinforced composites show a better tolerance of damage due to their smaller fibre diameters than the SiC-fibre-reinforced composites.

SiC- und kohlenstoffaserverstärktes Glas unter Biegespannungs-Wechsellastbeanspruchung

Wechselbiegespannungsexperimente werden an faserverstärkten DURAN-Glas-Verbunden durchgeführt, wobei Nicalon NL 202-SiC-Fasern und zwei verschiedene Kohlenstoffasern, eine hochfeste Faser (ht) und eine Hochmodulfaser (hm), zur Anwendung kommen. Es werden sowohl der Einfluß von Verbundwerkstoffen mit homogener und inhomogener unidirektionaler Faserverteilung als auch der von Verbundwerkstoffen mit bidirektionaler 0°/90°-Schichtenfolge untersucht. Im Gegensatz zu vorangegangenen wegegesteuerten Experimenten zeigen die vorliegenden spannungsgesteuerten Versuche nach einer Vorbehandlung von 1000 Zug-Druck-Wechsellastzyklen Ermüdung (beginnende Schädigung) bereits bei Lastamplituden unterhalb der Elastizitätsgrenze der nicht vorbelasteten Proben (unterhalb der Bendover- oder Knickspannung). Trotz dieses Unterschiedes wird auch hier der „Trainingseffekt" der vorangegangenen (wegegesteuerten) Untersuchung gefunden, d. h. ein Anstieg der Knickspannung mit zunehmender Spannungsamplitude nach 1000 Wechsellastzyklen. Die kohlenstoffaserverstärkten Verbundkörper zeigen generell eine bessere Schädigungstoleranz auf Grund ihrer kleineren Durchmesser als die mit SiC-Fasern verstärkten Verbundkörper.

1. Introduction

The goal of development of modern high-performance materials is not only to obtain high strength and high fracture strain but also to reach a large tolerance of damage. The elastically stored energy of a material under load is proportional to the product of stress and strain within the linear range [1]; the stronger the material the larger is this energy and the more catastrophical may be the course of fracture.

The experimentally determined strength of a material is basically lower than its theoretical strength. The reason is the presence of inherent voids, structural defects, flaws, cracks and microcracks, which give rise to local excessive stress peaks leading to unstable crack growth [2 to 4]. An increase in damage tolerance may be obtained, if the local excessive stress can be prevented or reduced and if the crack growth which would lead to the macroscopic fracture of the material is shifted to relatively larger fracture stresses. A material behaves tolerant at damage when the course of fracture is not catastrophical, i.e. when the stored elastic energy at fracture is released slowly under further increase of strain.

Plastic deformations at the crack tip in metals cause a release of excessive stress by the movement of dislocations and gliding layers. Mainly the metallic bonding character is the origin for the large ductility of these materials, which is lacking in ceramics and glasses with their covalent and ionic bonding types. Therefore, the fibre reinforcement of these brittle materials has been proved to be successful [5 to 8].

Fibre-reinforced glasses exhibit fracture strength values being up to a factor of 15 larger than those of the bulk glass depending on the fibre type and test method. After crossing the maximum fracture stress the elastic energy is destructed by energy-consuming effects such as debonding, pull-out and delamination preventing or reducing at least a catastrophical fracture [9 to 11].

It is very important for the practical application of these materials that the favourable mechanical prop-

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erties are preserved also after repeated cyclic loadings, i.e. to have good durability and long lifetime. Investigations on the fatigue of fibre-reinforced glasses were made usually in cyclic tensile tests or in cyclic bending tests [12 to 16]. The tested composites in those investigations, SiC- as well as carbon-fibre-reinforced glasses and glass-ceramics, show very good mechanical durability if the applied alternating-load amplitude is smaller than the so-called bendover stress of the composite. Beyond this critical stress value the composites show fatigue.

2. Object of the paper

Own investigations were performed recently [17] on SiC- and carbon-fibre-reinforced DURAN glass in cyclic bending load experiments, which differ in two important points from the just mentioned experiments [12 to 16]: First, the composites were tested in a bending regime with alternating tensile-compressive bending load (amplitudes between $+\sigma$ and $-\sigma$ crossing $\sigma = 0$) whereas in [12 to 16] the alternation of the amplitude was between $+\sigma$ and 0 or $-\sigma$ and 0, respectively. In [3 and 4] it is pointed to the fact that certain fatigue mechanisms in ceramic materials are activated under the former type of cyclic-load condition (amplitude between $\pm\sigma$ through $\sigma = 0$). Second, the own alternating bending-load experiments were strain-controlled, while those in [12 to 16] were stress-controlled.

The main results are that the pretreatment of 1000 loading cycles does not lead to any decrease of the mechanical properties as long as the load amplitude does not exceed the bendover stress. Particularly the carbon-fibre-reinforced glass behaves very tolerant during damage when the load amplitude is between bendover stress and bending strength of the composites. As a result of crack initiation and crack saturation the Young's modulus decreases only slowly but the bendover stress increases markedly as was measured after the cyclic pretreatment of the composites and which was called "training effect" in [17].

In the present paper alternating-load experiments are performed under the condition of stress reversal as described in [17], however, not strain- but stress-controlled in order to get an answer to the question whether the large damage tolerance obtained in the strain-controlled investigation is also found in stress-controlled experiments. This investigation includes SiC- and carbon-fibre-reinforced DURAN glass composites and in the case of carbon fibres high-tensile strength (ht) as well as high-modulus fibres (hm) are applied; the ht-fibre composites in different quality with respect to unidirectionally fibre distribution and in a bidirectionally 0°/90° manner.

3. Experimental

3.1. Preparation of the composites

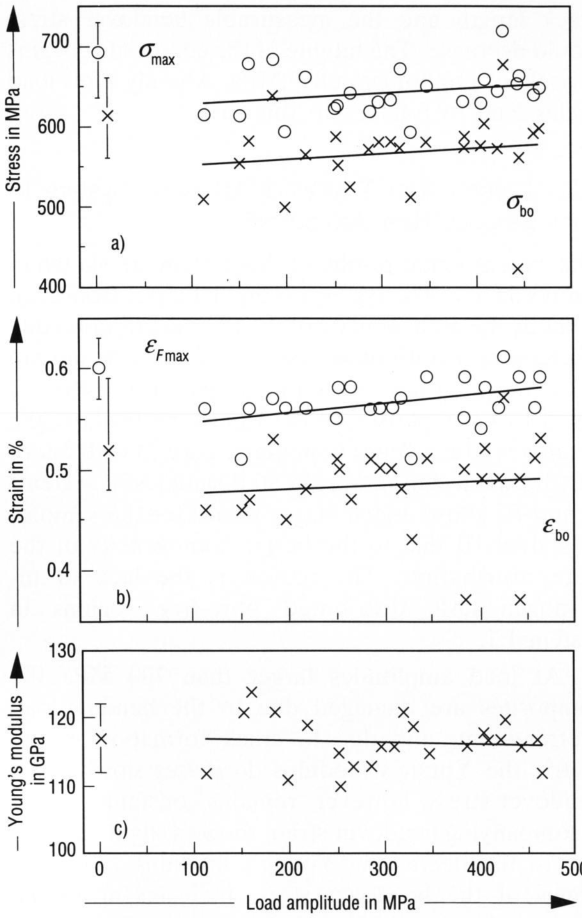
Five different fibre–glass systems were prepared by the sol-gel-slurry method with the subsequent steps winding, hydrolysis, polycondensation of the silicon-alkoxide solution, drying, hot-pressing of the prepregs and sawing of the composites to the dimensions $(95 \times 3 \times 4)$ mm^3. More information of preparation may be found in [17 to 19].

All samples have a fibre content of (40 ± 2) vol.%, the matrix is DURAN glass, a borosilicate glass from Schott Glaswerke, Mainz (FRG). The samples of system I contain SiC fibres Nicalon NL 202, (Nippon-Carbon, Tokyo (Japan)) in the samples of systems II and III carbon fibres Toray T 800 HB (Toray-Industrie Deutschland GmbH, Frankfurt/M. (FRG)) are introduced, system III samples exhibiting a more homogeneous fibre distribution than those of system II due to preparation. The composites of system IV are bidirectionally reinforced in 0°/90° ply by the Toray carbon fibres T 800 HB. The composites of system V are equipped with unidirectionally oriented hm-carbon fibres Toray M 55 J.

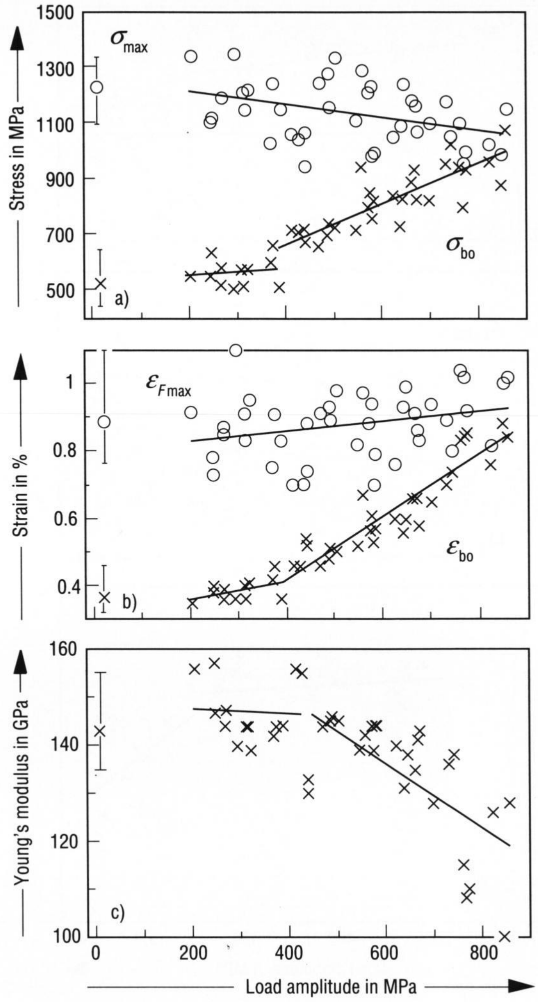
3.2. Procedure of the alternating bending load experiments

The composites were pretreated by a sinusoidally alternating tensile and compressive stress-strain bending loading on each side of the samples at 1 cps as is described in section 2. and in [17], however, in a stress-controlled manner with the help of a servo-hydraulic testing machine (MTS Systems GmbH, Berlin (FRG)). The same reversible three-point equipment as described in [17] could be used. The span was 75 mm, thus, a ratio of this length, l, to sample height, d, was maintained to be $l/d > 20$ [20 and 21]. The stress-strain diagrams were obtained with the same equipment at a strain rate of 1 cm/min. Each pretreatment of the composites includes 1000 load cycles at a certain constant stress amplitude. The largest alterations of the mechanical properties due to cyclic loadings happen after the first 100 to 1000 cycles as was found in the authors' experiments and in [15]. One period of 1000 load cycles needs about 17 min at a frequency of 1 cps. Within the frame of the present experiments the main point of this study is to investigate a large number of samples in order to present rather good statistics than a larger number of load cycles for single samples. The force-time runs were recorded by a signal memory recorder and evaluated and plotted by an HP 9000 computer (Hewlett Packard GmbH, Böblingen (FRG)).

After these pretreatments the mechanical properties of the composites were tested with a universal testing machine from Zwick, Ulm (FRG), (type 1455). The comparison is to be made between



Figures 1a to c. Bending strength, σ_{max}, and bendover stress, σ_{bo}, (figure a), bending strains at fracture, ε_{Fmax}, and at bendover stress, ε_{bo}, (figure b), and Young's modulus (figure c) as a function of the preloading amplitude after 1000 load cycles for system I. At the left-hand side of the diagram (also in the following figures): mean values and scatter bars of measurements of 15 non-pretreated samples are shown.



Figures 2a to c. Bending strength, σ_{max}, and bendover stress, σ_{bo}, (figure a), bending strains at fracture, ε_{Fmax}, and at bendover stress, ε_{bo}, (figure b), and Young's modulus (figure c) as a function of the preloading amplitude after 1000 load cycles for system II.

non-pretreated and differently pretreated samples with respect to the bendover stress, the bending strength and the Young's modulus.

4. Results

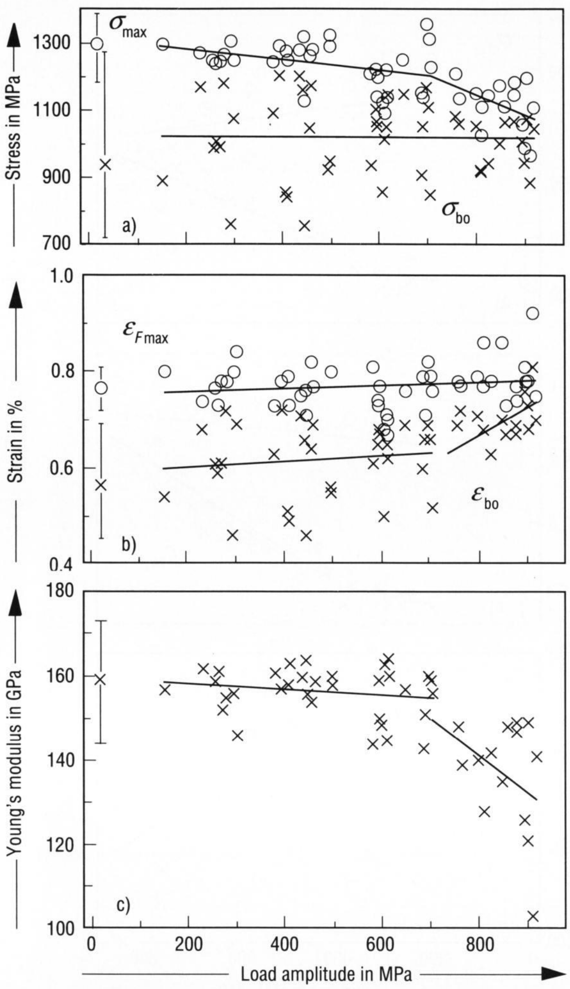
4.1. SIC fibre/DURAN glass (system I)

The bending strength, bendover stress and the Young's modulus of the composites of system I are demonstrated in figures 1a to c after the pretreatment of 1000 load cycles as a function of the load amplitude. On the left-hand side of these figures and in all the following figures the values of the unpretreated samples are given which are mean values of 15 samples. The error range indicates the maximum and minimum values. The bendover stress is that stress at which the first nonlinearity occurs in the stress-strain diagram produced by the formation of the first microcracks in the matrix.

At load amplitudes larger than 450 MPa a complete damage of the samples is registered which happens already after about 100 load cycles. Those values are not shown in figures 1a to c, only those which survived 1000 load cycles. Below this sharp limit of the load amplitude there is seen no influence of the pretreatment of 1000 load cycles on the mechanical properties because the values are within the limit of the non-pretreated samples.

4.2. Carbon fibre T 800/DURAN glass (system II, inhomogeneous fibre distribution)

Figures 2a to c show the course of the stresses, strains and of the Young's modulus for the samples of this system. Load amplitudes larger than 400 MPa cause microcrack formation within the matrix as is indicated by the decrease of the Young's modulus but at nearly unchanged bending strength, σ_{max}, and fracture strain, ε_{Fmax}; a very slight deviation is within the scatter of the measurements. These two latter properties are determined mainly by the fibres and indicate that the fibres themselves are not seriously



Figures 3a to c. Bending strength, σ_{max}, and bendover stress, σ_{bo}, (figure a), bending strains at fracture, $\varepsilon_{F\max}$, and at bendover stress, ε_{bo}, (figure b), and Young's modulus (figure c) as a function of the preloading amplitude after 1000 load cycles for system III.

damaged by the alternating bending load cycles but only the matrix.

With respect to the damaged matrix two statements may be made: First, the nearly constant bending strength indicates that the matrix still transfers the load to the fibres despite of the produced microcracks. Second, a further consequence of the microcrack formation is an increase of the bendover stress of the stronger pretreated samples ("training effect").

The microcracks obviously appear at the weakest points of the composites, i.e. mainly at those regions where the local fibre concentration is relatively small (inhomogeneous fibre distribution). Further microcracks are formed only at stresses during further loadings which are larger than the preloading amplitude. This behaviour indicates a microcrack-insensitive behaviour of this composite system. If the behaviour were be microcrack-sensitive, the crack would continue to grow at each load cycle, the excess stress at the crack tip would increase with increasing crack length and the measurable bendover stress would decrease. The fatigue of the composites begins at preload amplitudes at 850 MPa. Already a few load cycles lead to fracture in this case.

4.3. Carbon fibre T 800/DURAN glass (system III, homogeneous fibre distribution)

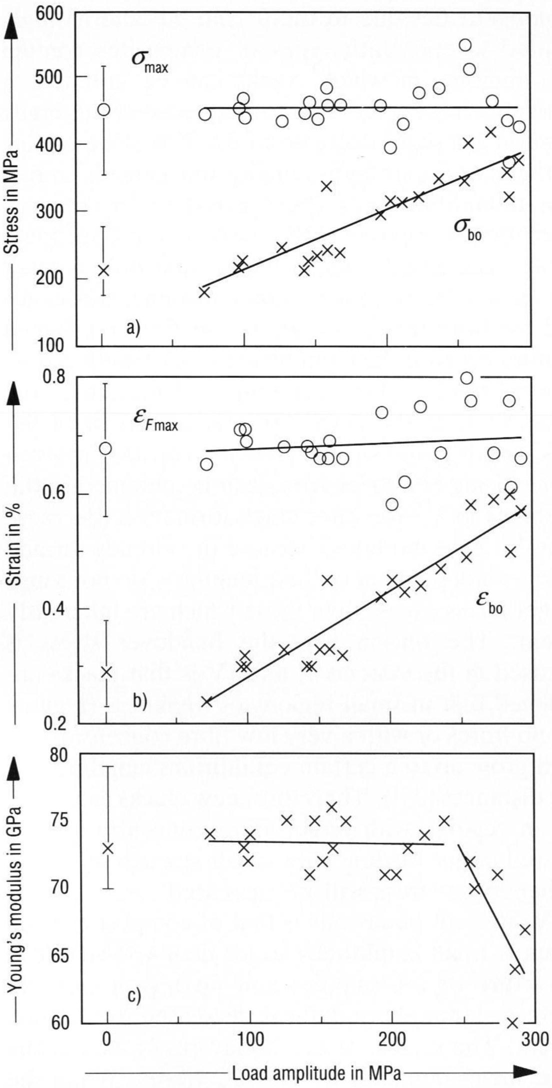
The experimental results of this system are shown in figures 3a to c. The systems II and III differ from each other by the homogeneity of the fibre distribution due to preparation differences (see e.g. figures 2b and c in [17]); the total fibre contents are identical ((40 ± 2) vol.%). A comparison of the values, particularly the bendover stress values (compare figure 2a with figure 3a), between the non-pretreated samples of systems II and III shows much larger values for the samples of system III due to the better homogeneity of the fibre distribution. The reason is the lack of inhomogeneously distributed fibre-free regions in system III.

At load amplitudes larger than 700 MPa the composites are damaged due to the bending-load pretreatment, and due to crack formation in the matrix the Young's modulus decreases slowly. The bendover stress, however, remains constant and the accompanying bendover strain shows a slight increase due to the decreasing Young's modulus. The constancy of the bendover stress indicates again the microcrack insensitivity of the present fibre–glass system. Although cracks are initiated in the matrix after 1000 load cycles at amplitudes larger than 700 MPa (decrease of Young's modulus), no crack growth takes place at lower stress level than that of the original bendover stress; the created cracks do not act as defects. Large preloading amplitudes involve only a small decrease in bending strength, while $\varepsilon_{F\max}$ remains constant, thus, the strength values of the composites of system III are always larger than those of system II.

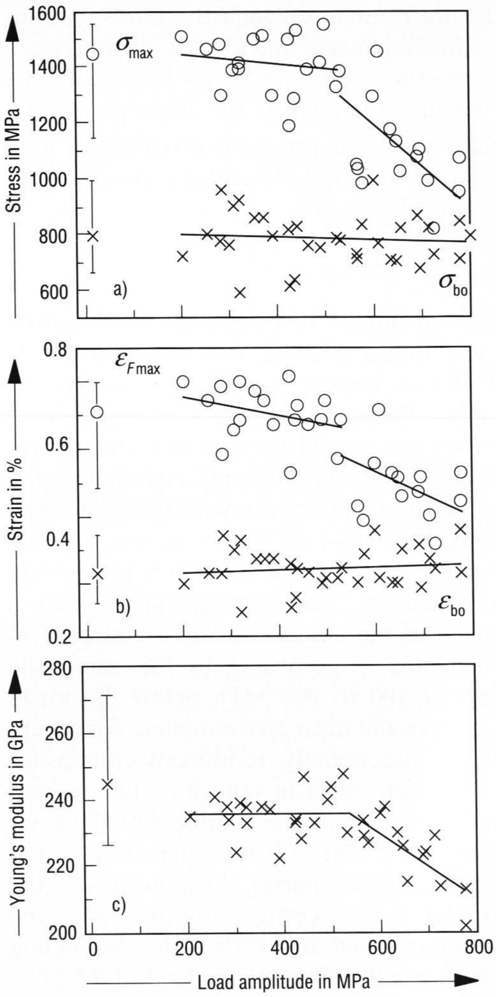
Load amplitudes larger than 900 MPa lead to a quick fatigue of the composites; the fracture happens after about 500 load cycles. Composites which have been pretreated with load amplitudes between 700 and 900 MPa show a slight separation (delamination) of fibres from the matrix at the points of largest tensile stresses.

4.4. Carbon fibre T 800-0°/90°/DURAN glass (system IV)

In contrast to the composites with unidirectional fibre reinforcement the bidirectionally reinforced samples are damaged not by fracture of the fibres on the tensile stress side under the three-point bending test but by delamination of single fibre layers. The problem of this delamination process is the most frequent origin of damage for laminate systems after [22].



Figures 4a to c. Bending strength, σ_{max}, and bendover stress, σ_{bo}, (figure a), bending strains at fracture, ε_{Fmax}, and at bendover stress, ε_{bo}, (figure b), and Young's modulus (figure c) as a function of the preloading amplitude after 1000 load cycles for system IV.



Figures 5a to c. Bending strength, σ_{max}, and bendover stress, σ_{bo}, (figure a), bending strains at fracture, ε_{Fmax}, and at bendover stress, ε_{bo}, (figure b), and Young's modulus (figure c) as a function of the preloading amplitude after 1000 load cycles for system V.

The results of this system are shown in figures 4a to c. The mean Young's modulus of the non-pretreated composites corresponds to about half of the values of samples with unidirectionally oriented fibres of the same type (compare system III, figure 3c with figure 4c). This means that the 90° layers in the bidirectional composites do not contribute to reinforcement or/and elasticity. Also the bending strength and fracture strain values are somewhat lower for the bidirectional reinforcement than those for the unidirectional composites due to the different modes of fracture. This is seen from the comparison of figures 3a and b with figures 4a and b.

From the presentation of figures 4a and b it is clear that already relatively low load amplitudes lead to an increase of the bendover stress and bendover strain. A decrease of the Young's modulus is registered only at larger pretreatment amplitudes (figure 4c). This points to the suggestion that microcracks are initiated at low load amplitudes in such regions which are not or only little concerned in taking over load, for instance the particular 90° layers or the 0°/90° interlayer. Load amplitudes larger than 300 MPa lead to delamination and to damage of the composites, a process which starts after about 100 load cycles.

4.5. Carbon fibres M 55 J/DURAN glass (system V)

The results of this system with the hm-carbon fibres are shown in figures 5a to c. Load amplitudes larger than 500 MPa lead to a decrease of all the measured properties. The decrease of the Young's modulus indicates microcrack formation in the glass matrix, the low bending strength and bendover stress values may be caused by the assumption that a part of the initiated matrix cracks damage the fibres in two ways:

First, cracks are running through the fibres without deflections (direct damage) and second, the fibres can be cut after microcrack formation by fractured glass pieces during new loadings at the three-point test.

A damage of the composites is produced by load amplitudes larger than 750 MPa after a series of load cycles of about 200 to 300.

5. Discussion

An important result of investigations in literature [12 to 16] was that a damage of composites is only possible if the cyclic-load amplitude exceeds the bendover stress. These investigations were made with load cycles from zero load to tensile and compressive loadings, respectively, only $(0,+\sigma,0,+\sigma)$ whereas the present investigations are made with both-side load cycles passing zero load $(0,+\sigma,0,-\sigma,+\sigma)$, i.e. the samples are stressed on both sides in a tensile and compressive manner. The consequence is that a partial damage of the composites occurs already at stresses which are – according to the composite system – about 100 to 300 MPa below the mean bendover stress of the untreated samples. An exception are the bidirectionally reinforced composites (section 4.4.) which differ in failure (delamination) on principle from that of the unidirectionally reinforced composites (failure of the latter ones on the maximum tensile stress parts). Composites of the systems I to III were investigated earlier with the same test apparatus and also with both-side loading cycles, but with strain-controlled loadings of the MTS machine. Strain-controlled cyclic loading means in this case that a decrease of the Young's modulus or a decrease in strength of a sample does not lead to a readjustment of the stress to the value at the beginning of the alternating-bending load as is the case for the stress-controlled cyclic loading. Therefore, an alteration of the mechanical properties could be detected under the condition of the strain-controlled cyclic pretreatment of the samples only above preloading amplitudes larger than the mean bendover stress of the composites whereas under the present investigation with stress-controlled cyclic loadings the demands are larger, thus, fatigue is faster and failure occurs at somewhat lower values than that of the bendover stress.

An interesting fact is the increase of the bendover stress towards larger preloading amplitudes of composites for the systems II and IV ("training effect"). This effect can be interpreted in the following way. A high fibre concentration in a composite is connected with a high bendover stress and therefore, with a high fracture stress of the glass matrix [23] and vice versa. Now, the composites of system II exhibit a very inhomogeneous fibre distribution due to preparation condition, i.e. regions with a high fibre concentration coexist with those of a low fibre concentration. In an analogous manner the composites of system IV show inhomogeneities due to the 0° and 90° alternatingly oriented layers. Both types of composites contain weak regions, in which cracks can be initiated at relatively low stresses. The consequence of this crack initiation is a slight decrease of the Young's modulus but the bending strength remains unchanged. Unlike as in monolithic glass (bulk glass) or in ceramics where the stress excess at the crack tip is proportional to the crack length, which means that only a small stress is needed to make a crack instable, this is not valid for fibre-reinforced glasses or fibre-reinforced ceramics because the equilibrium crack length is only a few fibre-fibre distances long and therefore, the stress excess at the crack tip is independent of the crack length [24]. For this reason a constant or even an increasing bendover stress can be obtained in the systems II to V even after crack formation (decrease of the Young's modulus) because the already formed cracks – independent of their lengths – do not cause a larger stress excess than those which are inherently present. The reason why the bendover stress is increased in the systems II and IV is that cracks are produced first in small regions of weakness (regions with no fibres or with a very low fibre concentration) which grow up to a certain equilibrium length (some fibre distances [24]). Therefore, new cracks can occur only in regions with larger fibre concentrations at renewed larger loading with larger stresses by which the bendover stress will be increased.

A different behaviour is that of composites from system I. Load amplitudes larger than 450 MPa lead to fracture of the samples and no region exists in which the Young's modulus is decreased to a certain amount. The reason of this behaviour is seen in the larger mean diameter of the SiC fibres. While the carbon fibres have a diameter of about 5 μm (both types of the used fibres), the diameter of the SiC fibres is about 15 μm. The fibre-fibre distances are proportional to that, these are larger for the SiC fibre reinforcement than for the carbon fibre reinforcement at equal fibre volume content. The equilibrium crack length is that of a few fibre-fibre distances after [24]. Thus, the cracks are longer in the composites of system I than in those of the systems II to V. Due to these cracks being larger by a factor three the matrix is not any more able to transfer the load from outside to stresses on the fibres after a certain number of load cycles and, thus, the samples fail.

The smaller diameter of the carbon fibres also results in the simple fact that the number of carbon fibres per volume unit is by a factor of 10 larger than that of the SiC fibres at comparable fibre volume content from which results a much larger fibre/matrix interfacial area. The consequence of a large interfacial area is a favourable action on processes such as crack branching, crack deflections and crack stops which are advantageous to keep the length of the cracks in the carbon-fibre/glass composites smaller than in the SiC-fibre/glass composites.

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