

Preparation of C-fibre borosilicate glass composites: Influence of the fibre distribution on mechanical properties

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Optimum conditions with respect to preparation-determined fibre distribution in borosilicate glass composites were investigated. Continuous C-fibre bundles were impregnated with glass powder in silicon alkoxide solution and wound in parallel to preregs which were hot pressed into unidirectional composites. The influence of the glass particle size during the impregnation of fibre bundles and during hot pressing on the homogeneity of the fibre distribution was of special interest, as well as the influence of pressure and temperature on the densification of the composites. Optimum conditions were related to optimum values in the bending strength of the resulting composites. Under the optimum hot-pressing conditions the fibre volume content was varied. It has been shown that the fibre distribution was much more homogeneous when fine-grained glass powder was used for impregnation. At high fibre volume concentrations of the composites the distribution was better than at low concentrations. High fibre concentrations were connected with fracture toughness of the sample, as shown by the three-point bending experiments, whereas samples with low C-fibre concentration showed brittle behaviour.

1. Introduction

Composite materials have become increasingly important. For two decades the composite principle has also been applied to the reinforcement of glasses, glass ceramics and ceramic materials. It has been possible to drastically improve the less favourable properties of these materials, such as brittleness, low fracture strength and low thermal shock resistance, while the favourable properties are preserved, such as good chemical resistance, wear and oxidation resistance.

The reason for the very low experimental strength and high brittleness of the non-metallic inorganic materials, in contrast to the theoretical strength, is the well-known crack formation, with those at the surface being the most critical. A three-axial stress condition acts at the crack tips leading to a stress excess with increasing load, which increases with the increasing root of the crack length (after Griffith). Hence, the stress at the crack tip is many times larger than the outer stress of the load [1]. When the critical stress intensity is exceeded the material fails catastrophically by unstable crack growth. The elastically stored energy of a loaded material is proportional to the product of stress and strain, thus, the stronger a material the larger the amount of this energy and the more catastrophic the fracture progress.

The brittleness of glasses is larger than that of ceramic materials because the cracks in the latter can be stopped, deflected or ramified at the grain boundaries. In order to minimize the stress excess the critical size of cracks needs to be kept as small as possible [2].

This means smoothing down the surface satisfactorily, for glasses by heat polishing or etching with hydrofluoric acid, for example [1]. However, this is not very effective and the improved surface is very sensitive to new crack formation. A much more effective way to improve the strength of glasses is by thermal or chemical hardening [1], in which compressive stresses are produced in the glass surface and in the crack tips of the surface flaws which are pressed together ("closed"). In this way the strength of glasses is increased by a factor of ca. 3–5, but brittleness, and therefore the catastrophic fracture behaviour, are also increased due to the increase in the stored elastic energy.

The incorporation of SiC or Si₃N₄ whiskers in glasses also leads to larger strength values, but not to an increase in toughness [8, 9].

It has become usual to incorporate small ZrO₂ particles in ceramics in order to increase strength as well as toughness by the effect of stress-induced transformation of tetragonal into monoclinic ZrO₂ [3–7]. This procedure has not been applied systematically to glasses. One reason may be that the best improvement of strength, as well as toughness, of glasses is achieved by reinforcement with continuous long SiC- and C-fibres [10], while short (discontinuous) fibres do not increase the strength but only the toughness in a moderate way [10–12].

In the 1970s Phillips and co-workers [10, 11, 14, 15] produced the first long C-fibre reinforced borosilicate glasses (Pyrex) and obtained bending strength values of up to 700 MPa, with good toughness properties,

with fibre volume concentrations of 40–51%. Prewo and co-workers [9, 12, 16, 17] prepared SiC- and C-fibre reinforced glasses and LAS glass ceramics (see also [13]). With Pyrex glass these authors reached bending strength values of ca. 1000 MPa for C-fibres and 800 MPa for SiC-fibres. Dawson [18] and Dawson *et al.* [19] give values for the bending strength of SiC-fibre reinforced Pyrex glass which so far have not been obtained experimentally: 1250 MPa at a fibre content of 50 vol % and 1000 MPa at 40 vol %.

The objective of the present work has been to investigate the influence of various distinct preparation parameters on the mechanical properties of the resulting composites. The system C-fibre T800HB/DURAN glass serves as a model system. It will be shown that, besides pressure and temperature during the high-temperature pressing procedure, and the volume concentration of the fibres, the mean grain size of the glass powder in the slurry at the beginning of the preparation is of particular importance.

The samples with their systematically varied and selected preparation procedures were tested using the three-point bending experiment. This test appeared to be meaningful and efficient not only because most kinds of practical loadings of a material are bending stresses and strains, but because it is very advantageous in the sample preparation and handling to measure the bending parameters as opposed to the tensile strength, especially during the state of development of a composite system such as the above mentioned model system [20, 21]. The authors are aware of the disadvantage of the bending test method because of the not quite unequivocal failure of the samples produced by the superposition of tensile, compressive and shear stresses [22]. The maximum stresses in the bending test are only within the marginal layers of the samples, thus, the effectively stressed volume is relatively small compared to the whole sample volume and limited to the region of the marginal fibres [23].

2. Experimental procedure

2.1. Sample preparation

The preparation of the prepregs was done by the sol-gel slurry method described in [24–26]. Before winding the fibre bundle on a hexagonal drum they

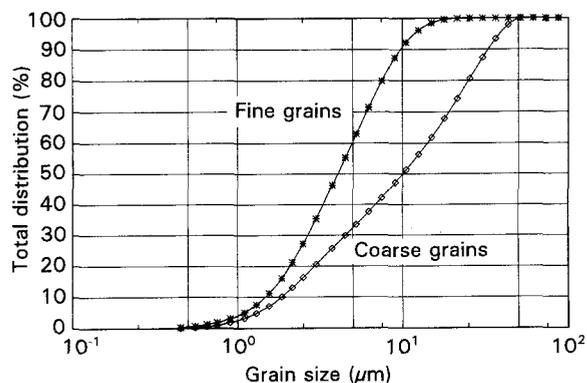


Figure 1 Total distribution of grain size of the DURAN glass powder used.

were drawn through a swirl-bath in which the fibres were loaded with glass powder and with a silicon alkoxide solution. This method has the advantage that the solution acts as a binder which forms a glass after polycondensation, hydrolysis and pyrolysis in contrast to the simple slurry method [14, 17].

The fibre volume content was determined by the ratio of glass powder to alkoxide solution. DURAN glass (Schott Glaswerke, Mainz) was used and the powder was prepared in two different mean grain sizes; their distribution is shown in Fig. 1.

The prepreg on the drum was heated then taken off the drum and cut into six quadratic prepreg plates ($10 \times 10 \times 1.5 \text{ cm}^3$). The very porous prepregs were densified by hot pressing in a graphite form which was heated inductively and pressed hydraulically. The composite samples were cooled under pressure. Decisive for the properties of the composites are the moment and the amount of pressure charge during heating up the samples, the final pressure value and the maximum temperature. These parameters were varied in this study.

The surface of the composite plates was ground by a pot grinder and cut into bars of $95 \times 3 \times 4 \text{ mm}^3$ (length \times height \times width) by a diamond saw. The three-point bending method was used and attention paid to the ratio $l:h$ ($l = 75 \text{ mm}$ scan width; $h =$ sample height) which was always larger than 20 [22, 27], thus, the shear component had no influence on the bending strength. The samples were oriented parallel to the direction of the high-temperature pressure procedure in such a way that the upper or lower surface of the plate was tested in tensile stress. The tests were done in a ZWICK-universal proof machine, type 1455, with a strain rate of 10 mm min^{-1} . The characteristic properties of the matrix glass and the high-strength C-fibres Toray T800 are listed in Table I.

3. Results

3.1. Effect of step-wise pressure increase and grain size of the glass powder

The temperature–pressure procedure and the viscosity of the DURAN glass are given in Fig. 2. A prepressure of 0.2–0.3 MPa was always present, which acted as a threshold value when the working piston contacted the pressure cell. The pressure increase had

TABLE I Characteristic properties of the matrix glass and T800 fibres

Glass	Young's modulus (GPa)	Density (g cm^{-3})	Thermal expansion coeff. (10^{-6} K^{-1})	T_g ($^{\circ}\text{C}$)	Working temperature ($^{\circ}\text{C}$) ($\eta \approx 10^3 \text{ Pa}$)
DURAN	63	2.23	3.25	530	1260

Fibre	Tensile strength (MPa)	Young's modulus (GPa)	Density (g cm^{-3})
T800	5490	294	1.81

TABLE II Properties of composites from coarse and fine grains in the sol-gel slurry

Sample	Bending strength, σ_{max} (MPa)	Bendover stress, σ_{bo} (MPa)	Young's modulus (GPa)
Coarse grain, no pressure	801 ± 39	433 ± 45	131 ± 4
Coarse grain, prepressure	1107 ± 68	494 ± 93	142 ± 12
Fine grain, prepressure	1218 ± 86	905 ± 214	146 ± 7

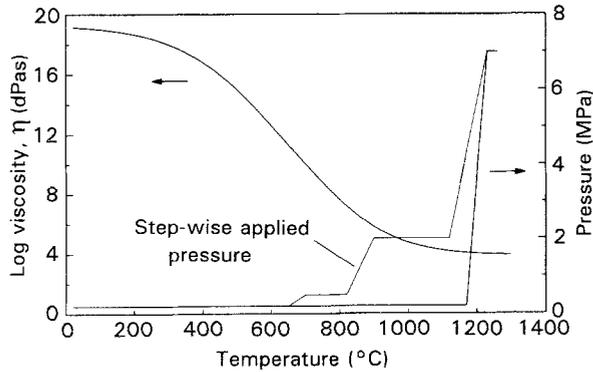


Figure 2 Pressure-temperature program for composite preparation and viscosity-temperature curve of the DURAN glass matrix.

to be slow enough to avoid damage or misorientation of the fibres.

In Table II selected mechanical properties are listed from bending tests of samples which were prepared under different conditions. All composites have a fibre volume content of 40 ± 2 vol %, the maximum (final) pressure was 7 MPa and the maximum pressing temperature 1260 °C. "Prepressure" means that the pressure was increased step-wise according to Fig. 2.

Fig. 3a and b give an impression of the fibre distribution over the cross-section of these composites. The inhomogeneous distribution belongs to a sample prepared with coarse-grained glass powder and densified without prepressure, the homogeneous distribution of a sample prepared with fine-grained glass powder and densified with prepressure.

In order to analyse the fibre distribution in a quantitative way, a circle of 80 μ m diameter was chosen and the fibre concentration of this area studied. In a series of circles the fibre concentration was determined. Due to the statistical distribution there were circles with more or less fibres within the whole cross-section of a composite. In total 300 circles were measured for each of the three different composites, listed in Table II (Fig. 4). It is seen that the fibre distribution with the smallest half-maximum width is obtained due to the application of a prepressure during hot pressing and due to the use of fine-grain glass powder. This means greater homogeneity in fibre distribution. Two contradictory advantages are connected with this: (1) fibre-poor or fibre-less regions within a composite, in which already low stress matrix cracks can occur, are avoided; (2) the number of fibre-fibre contact points within fibre-rich regions, which are avoided here, is large. Fibre-fibre contact points are acting as centres of increased stress concentration [28], i.e. cracks may propagate from contact point to contact point throughout the composite.

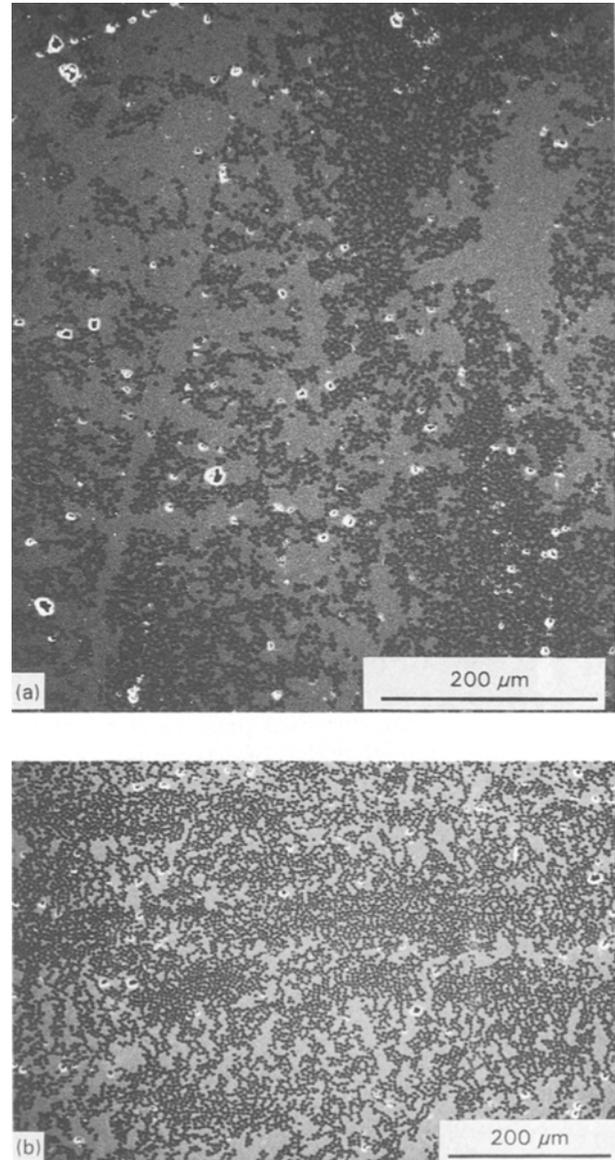


Figure 3 Micro-structure of a C-fibre T800/DURAN glass composite, $V_f = 40$ vol %. (a) Coarse-grained slurry without prepressure. (b) Fine grained slurry with prepressure.

The reason for the very homogeneous fibre distribution when fine-grained glass powder is used may be that the grain size best fits the space between the fibres (wedges) of the fibre bundles, thus, the fibre bundle will be better infiltrated by the slurry, that means, the matrix is already distributed more homogeneously in the preregs.

The step-wise increased prepressure causes a more regular distribution of the glass melt due to a moderate pressure at higher viscosities over a longer duration, but also the separation tendency between glass

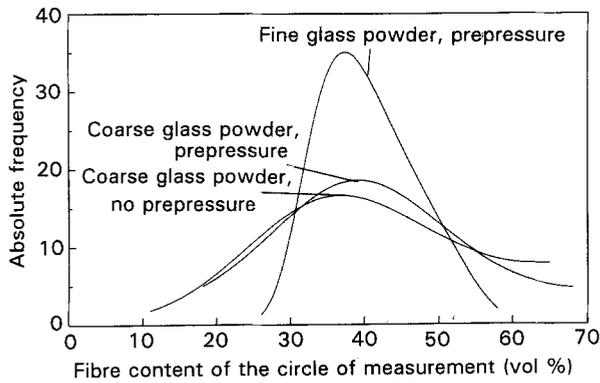


Figure 4 Fibre distribution (absolute frequency versus fibre content in a circle of 80 µm diameter) of composites with $V_f = 40$ vol % after application of various process parameters.

melt and C-fibres (due to low or no wetting) is counteracted by the prepressure. It is important that the prepressure is applied above the glass transition temperature (T_g) of the glass to avoid local damage by the sharp-edged glass grains below T_g .

The experiments described in this section have the consequence that fine-grained glass powder is used in the following sections for the preparation of C-fibre/DURAN glass composites and the densification procedure with prepressure.

3.2. Influence of pressing temperature

Fig. 5 shows the strength and the densification of two composites with different fibre concentrations versus pressing temperature, the final pressure was 7 MPa. The maximum strength of the composites with the two fibre concentrations was obtained at a pressing temperature between 1250 and 1300 °C.

Pressing temperatures which were too high caused an increase in the distribution width of the measured values, an indication of the beginning of degradation of the fibres (thermally) and/or reaction with the glass melt (chemically), and a disturbance of the fibre architecture of the composites caused by partly pressing out the matrix of the pressure cell.

The densification of the composites increased with increasing temperature. The Toray T800 C-fibre as a high-strength fibre contained a "rest" amount of nitrogen which was released during the high-temperature pressing, and the surface of the fibre was very rough due to the fibre drawing process. Both facts caused the increase in densification with temperature: the release of nitrogen decreased and the glass melt infiltrated more and more into the fine surface roughness with decreasing viscosity.

3.3. Influence of pressure

The effect of compressive stress on the bending strength and on the densification of C-fibre T800/DURAN glass composites is shown in Fig. 6. The fibre concentration is about 50 vol %. The preparation was carried out under optimum conditions concerning the treatment of the slurry, the prepressure and the final pressing temperature. The largest

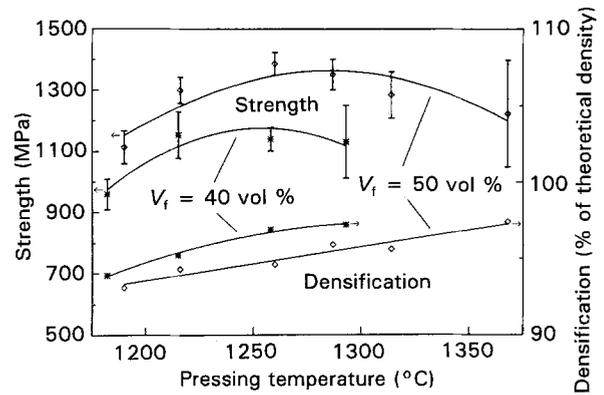


Figure 5 Bending strength and densification of composites with 40 and 50 vol % fibre content versus pressing temperature, final pressure 7 MPa.

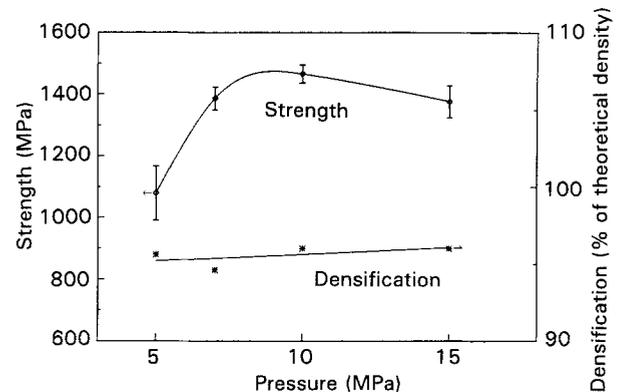


Figure 6 Strength and densification of composites with 50 vol % fibre content versus pressure at a pressing temperature of 1260 °C.

strength values were obtained at pressures ca. 10 MPa. Only slightly lower strength values were observed at 7 MPa, but at 5 MPa the values were significantly lower. It appeared opportune to reduce the pressure from 10 to 7 MPa in some cases of lower fibre content with respect to the longer lifetime of the graphite pressing form and with respect to the partly pressing-out of the matrix. The densification increased only negligibly with increasing pressure. It is assumed that the pressure primarily influenced the degree of the mechanical contact at the fibre-matrix interface, i.e. the degree of penetration of the glass matrix into finest surface roughness. This explains the density of the composites increasing very slowly, and being overlapped largely by the scatter of the measured values.

3.4. Influence of the fibre volume content

The prepregs of this series were hot pressed at 1260 °C at a pressure of 7 MPa. The bending strength and the bendover stress of the composites are plotted versus fibre content in Fig. 7a. The bendover stress is the stress value beyond that where the stress-strain diagram shows non-linearity. The two properties show maxima at a fibre concentration ca. 50 vol %.

The Young's modulus increases with fibre volume content (Fig. 7b). The experimentally determined Young's modulus almost coincides with the theoretically calculated one on the basis of the linear mixing rule (LMR) within the range of 35–55 vol %. This

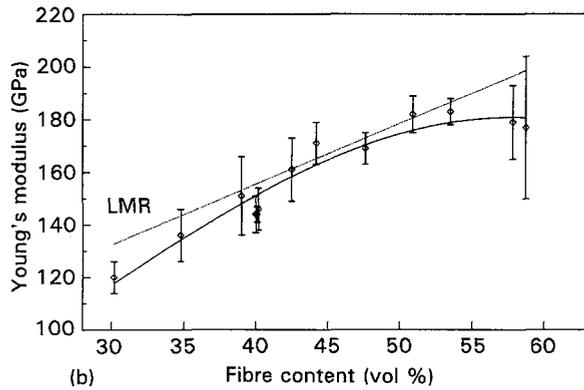
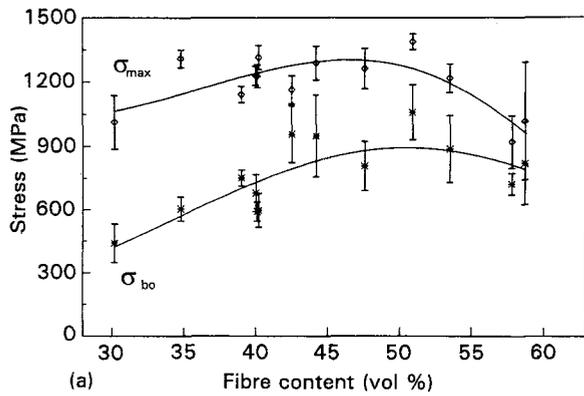


Figure 7 Bending strength and bendover stress (a) and Young's modulus (b) versus fibre content, final pressure 7 MPa, pressing temperature 1260°C. LMR-curve calculated by the linear mixing rule.

indicates that the utilization of the fibres is best in this range.

Two effects arise with increasing fibre volume content which lead to opposite influences on the mechanical properties: first, the number of fibre-fibre contacting points increases which produce cracks and local stress excess; second, the fibre distribution becomes more regular (homogeneous). In Fig. 8 the fibre distributions related to the measured areas of 80 μm diameter are demonstrated for various fibre concentrations. The improvement of the homogeneity in the fibre distribution with increasing fibre content leads to a drastic increase primarily in the bendover stress (Fig. 7a). An irregular distribution of fibres is connected to larger regions with relatively low fibre concentrations within the composites, in which cracks may have been initiated and propagated at low stresses. The increase in the number of fibre-fibre contact points with increasing fibre concentration has more influence on those mechanical properties of the composites which depend primarily on the fibre properties. This is the bending strength of the composites which is determined mainly by the fibre tensile strength while the highly crack-damaged matrix has the task only to fix the fibres locally. From Fig. 7a it follows that the bending strength of the composites increases only slightly when the fibre concentration is increased from 30 to 50 vol %. In other words, the fibre utilization with respect to strength becomes smaller and smaller by increasing the number of contact points.

The toughness of the composites beyond the strain at maximum fracture stress also increases with in-

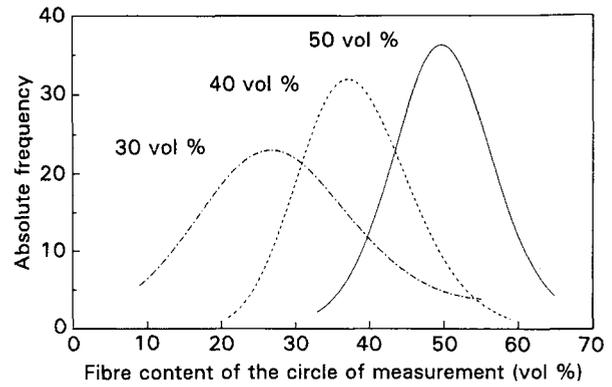


Figure 8 Fibre distribution (absolute frequency versus fibre content in a circle of 80 μm diameter) at various fibre concentrations; 30, 40 and 50 vol % in the composites.

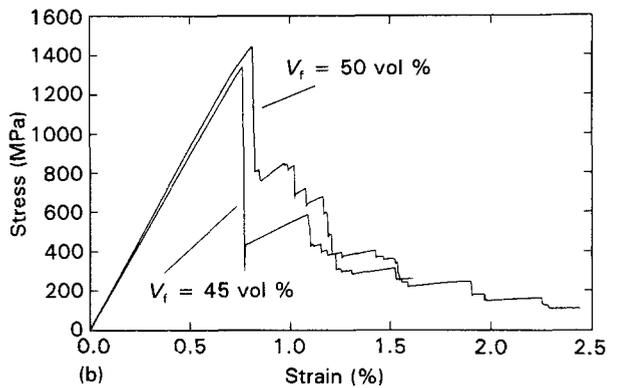
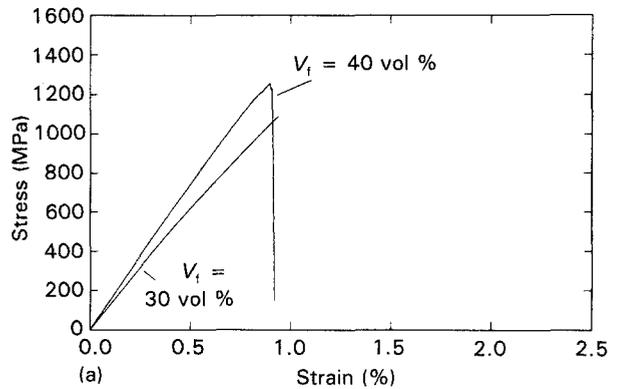


Figure 9 (a) and (b) Stress-strain diagrams of composites with various fibre concentrations.

creasing fibre content (Fig. 9a and b). This toughness in the bending test is due to the delamination phenomena. The tensile stress in the marginal fibre layers becomes larger than the fracture stress in these regions, fibres break and the cracks through the matrix, originally perpendicularly to the fibres, are deflected parallel to and along the interface of the fibre matrix; Crack branching also occurs. Fracture energy is consumed in this way. The crack will be stopped before the whole cross-section of the composite is separated if the fibre concentration and the fibre-matrix interfaces are large enough. It is opportune for the process of crack stopping when the crack is intensively branched. The consequence of crack stopping is that the normal stresses within the still undamaged part of the composite increase, leading to a step-wise decrease and re-increase of the stress in the stress-strain diagrams beyond the strain at maximum fracture stress.

In composites with low fibre content the crack can propagate quickly, especially within the low fibre concentrated regions because it will either not be or will barely be branched. Therefore the composite will be split into two parts before the normal stresses can increase again. SEM micrographs of fracture surfaces at the tensile stress side of the bended samples are shown in Fig. 10a and b. The composite in Fig. 10a had a fibre content of 30 vol %, that in Fig. 10b 40 vol %. In both figures it can be seen that a great deal of fibres are broken close to the matrix surface. This means that the bonding between fibre and matrix is relatively strong and that the pull-out effect contributes only a small amount to the toughness of the composite. Concerning the free ends of the fibres in Fig. 10a and b, the glass matrix was broken off during

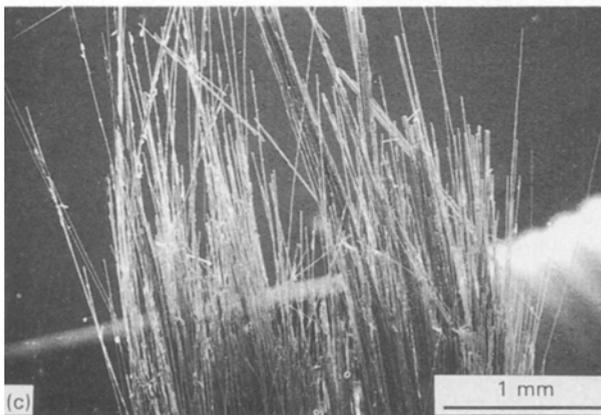
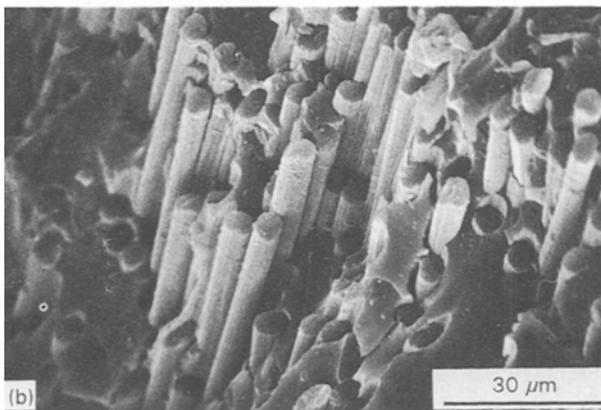
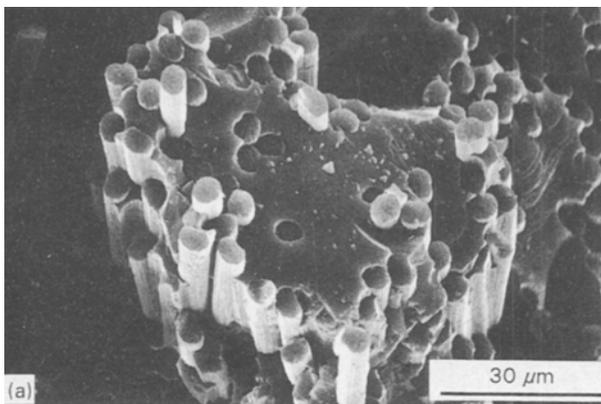


Figure 10 Fracture surfaces of composites at the tensile side of the bending test with various fibre concentrations, (a) 30; (b) 40; (c) 50 vol %.

crack propagation and crack branching. In Fig. 10c the fracture surface of a composite with a fibre content of 50 vol % is shown. In contrast to Fig. 10a and b the "surface" consists of fibre bunches with only few and small matrix pieces. In this case intensive crack branching took place due to the large fibre concentration, the result being a large toughness combined with a high strength (see Fig. 9b).

4. Discussion

The variation of preparation parameters of prepregs and composites as well as the pre-selection of various fibre volume concentrations have shown that the fibre distribution within the composites have great influence on their properties. The application of fine-grained glass powder and of prepressure lead to a distinct improvement in fibre distribution. This again lead to an improvement of the mechanical properties of the composites in the bending test, as is shown by typical examples in Section 3.1.

Fig. 11 demonstrates the statistical Weibull plots of samples which have been prepared by different procedures. Each system is based on at least 12, maximum 16, separate composite plates from which five samples per plate were tested. The fibre content was 40 ± 2 vol %. The Weibull evaluation was performed as a linear regression analysis in a two parameter presentation [29]. For the mean stress, σ_0 , and for the Weibull parameter, m , the following values are obtained:

coarse grained, no prepressure: $\sigma_0 = 755$ MPa,
 $m = 7.7$

coarse grained, prepressure: $\sigma_0 = 1139$ MPa,
 $m = 11.1$

fine grained, prepressure: $\sigma_0 = 1261$ MPa,
 $m = 19.6$.

Obviously, grain size has a large influence on m , whereas the prepressure largely influences σ_0 .

With respect to the effect of prepressure, no similar effects are reported in the literature. Usually, only approximate values or examples are given for maximum applied temperature and pressure. Possibly the publication of details was suppressed for special reasons. However, it cannot be excluded that chemical

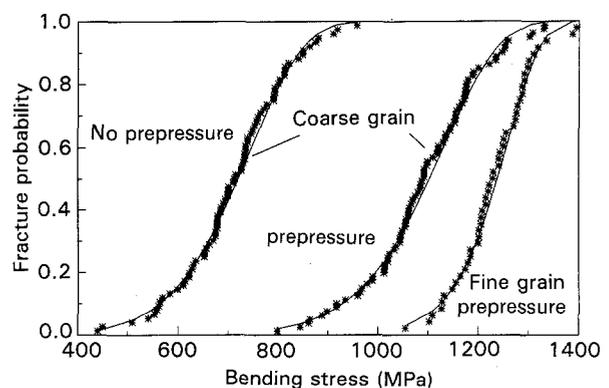


Figure 11 Weibull analysis of composites C-fibre T800/DURAN glass with 40 vol % fibre content after different preparation parameters which determine the fibre distribution.

reactions at the fibre-matrix interface are influenced by the application of prepressure because pressure dictates the contact between fibre and glass melt as well as the partial pressure of the gaseous reaction products, e.g. CO₂ [30].

Sambell *et al.* [14] compared two glass powders of different grain sizes by means of the properties of the resulting C-fibre/Pyrex glass composites. The two glass powders had a mean grain size of 10 µm and 40 µm with a large amount of fine-grained portions of 1 to 2 µm and ca. 5 µm. Better mechanical properties of the composites were obtained with the fine-grained powder analogous to these experiments. This effect is not explained in [14], only the bending strength values are compared with each other.

Other results were obtained by Bacon and Prewo [17]. A fine grinding process of the glass powder (100 h in a sphere mill) lead to a decrease of the fibre volume content and of the mechanical properties of the composites. No composites of equal fibre concentration were compared, no information about the grain size of the milled glass powder and no explanation for the described effects are given.

In another publication [11] it is argued that a large portion of glass particles smaller than the fibre diameter have to be present in the slurry in order to get a good distribution of fibres in the composite. This result can be confirmed by these experiments, not only for the C-fibre/DURAN glass composites but also for those with the bigger SiC-fibres (ca. 15 µm diameter) for which the fine-grained glass powder has shown no influence on the fibre distribution in the composite or on the properties.

The range of pressing temperature is for C-fibre/Pyrex glass composites in reported in the literature, e.g. [11, 17, 31, 32], cover 850–1300 °C, and a pressure range of 4–27.6 MPa. Agreement can be found in the results that the influence of pressing temperature on the resulting properties of the composites is larger than that of the pressure because the viscosity of the glass matrix depends exponentially on temperature. Bacon and Prewo [17] reports an increase of pressure from 13.8 to 27.6 MPa, at constant pressing temperature (1200 °C), caused a slight improvement of the mechanical properties of the composites, a deterioration was observed by Phillips [11] when the pressure was increased from 6.3 to 10.5 MPa.

5. Conclusions

The results in the literature and here show that not only are the preparation parameters of the high-temperature pressing process important for the mechanical properties of the composites, but also the parameters of the prepreg preparation, especially the perfection of the impregnation of the fibre bundles, the mean grain size, their distribution and the grain form (fibre damage). Beyond that it is important to know the mutual influences of room temperature, the elevated and high-temperature part of preparation parameters and their dependence on the various properties of the composites. In particular, it is shown in the present investigation that the application of pre-

pressure in the elevated temperature region and the preparation-dependent fibre distribution has a large influence on the composite properties and that the homogeneity is directed largely by the prepressure and by the milling process of the glass powder to a mean size of the order of magnitude of the wedges between the fibres.

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