Original Paper

Thermal expansion behaviour of fibre-reinforced DURAN glass

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The thermal expansion coefficients of DURAN glass reinforced by SiC and various carbon fibres are determined in order to investigate the influence of the different types of fibres and the preparation parameters on the thermal expansion behaviour of the composites. The coefficient of thermal expansion decreases with increasing Young's modulus of the fibres and with increasing fibre volume content. Composites with high-modulus carbon fibres and/or with high fibre volume concentration show a transition from contraction at lower temperatures to expansion at higher temperatures. "Zero expansion composites" can be produced for certain temperature intervals by selection of the fibre types and the fibre volume concentration. The experimentally determined expansion coefficients are compared with the theoretically calculated ones by the mixing rule. The agreement is very good for the high-modulus fibres, less good for the high-strength fibres.

Thermisches Ausdehnungsverhalten von faserverstärktem DURAN-Glas

Die thermischen Ausdehnungskoeffizienten von mit SiC- und verschiedenen Kohlenstoff-Fasern verstärktem DURAN-Glas werden bestimmt, um den Einfluß der unterschiedlichen Fasertypen und der Herstellungsparameter auf das thermische Ausdehnungsverhalten der Composites zu untersuchen. Der thermische Ausdehnungskoeffizient nimmt mit zunehmendem E-Modul der Fasern und mit zunehmendem Faservolumengehalt ab. Verbundwerkstoffe mit hochmoduligen Kohlenstoff-Fasern und/oder mit hohem Faservolumengehalt zeigen einen Übergang von Kontraktion bei tieferen Temperaturen zu Expansion bei höheren Temperaturen. "Nullausdehnungs-Composites" können für bestimmte Temperaturintervalle hergestellt werden durch Auswahl der Fasertypen und der Faservolumenkonzentration. Die experimentell bestimmten Ausdehnungskoeffizienten werden mit den aus der linearen Mischungsregel berechneten verglichen. Die Übereinstimmung ist sehr gut für die Hochmodulfasern, weniger gut für die hochfesten Fasern.

1. Introduction

Glass as a material of application exhibits a series of valuable properties, such as hardness, chemical resistance, wear resistance and very good optical and electrical properties. Very disadvantageous are the brittleness, the low thermal shock resistance and the low mechanical strength which prevent the application of the oxide glasses as a structural material for engineering construction in many cases. When fibre reinforcement is used these disadvantages may be changed to positive properties [1 to 10], high strength, reduction of brittleness and very good thermal shock resistance [11].

In particular successful is the reinforcement of glass with SiC and carbon fibres in a unidirectional or bidirectional (0°/90°) ply manner by which the composites show anisotropic or quasi-isotropic behaviour, respectively, with respect to their mechanical and thermal properties.

One of the most important thermal properties is the coefficient of thermal expansion and the most suitable one is the mean linear thermal expansion coefficient, $\alpha_{20/x}$, from room temperature, 20 °C, to temperature T_x which means the temperature dependence of the mean linear expansion coefficient. Among the large variety of glasses a separation is made by the amount of the coefficient of thermal expansion between the so-called "hard glasses" with $\alpha_{20/400} < 6 \cdot 10^{-6}\,\mathrm{K}^{-1}$ and the "soft glasses" with $\alpha > 6 \cdot 10^{-6}\,\mathrm{K}^{-1}$ indicating that α is a characteristic property with respect to mechanical and chemical properties of glasses [12].

In order to reinforce glass and to make this brittle material more tough, the Young's modulus, the strength and the strain of fracture of the fibres have to be larger than those of the glass and this is the case of SiC and carbon fibres. In connection to that the thermal expansion behaviour is strongly determined by that of the fibres. Heating up the carbon fibre-reinforced glass composites from room temperature a contraction is observed first, then an expansion [13]. By an aimed combination of carbon fibres with a slightly negative thermal expansion coefficient and of a matrix with positive coefficient of thermal expansion one can obtain a "zero expansion composite" with respect to a certain temperature interval and a certain fibre volume concentration [14].

The Linear Mixing Rule (LMR) can be applied to estimate the effective longitudinal expansion coefficient of the composites in which the Young's moduli have to be regarded, too [15]:

$$\alpha_{\mathrm{comp}} \equiv (\alpha_{\mathrm{f}} V_{\mathrm{f}} E_{\mathrm{f}} + \alpha_{\mathrm{m}} V_{\mathrm{m}} E_{\mathrm{m}}) / E_{\mathrm{comp}} \, .$$

According to [15] the agreement between the calculated expansion coefficients by this relation and the experimentally determined ones should be a measure for the

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Table 1. Characteristic properties of the applied carbon fibres and of the SiC fibre Nicalon NL202 (producer's data)

fibre type	tensile strength in MPa	Young's modulus in GPa	strain at fracture in %	density in g/cm^3	fibre diameter in mm	expansion coefficient in 10^{-6} K^{-1}
T300	3530	230	1.5	1.76	7	0.3
T800	5490	294	1.9	1.81	5	0.1
T1000	7060	294	2.4	1.82	5	0.1
M40J	4410	377	1.2	1.77	6	0
M46J	4210	436	1.0	1.84	5	−0.7
M55J	3920	540	0.7	1.93	5	–
M60J	3920	588	0.7	1.94	4.7	−0.9
M30	3920	294	1.3	1.70	6.5	–
M40	2740	392	0.6	1.81	6.5	−1.2
Nicalon	2750	189	1.4	2.55	15	1 to 2

Table 2. Properties of DURAN glass

Young's modulus in GPa	63
density in g/cm^3	2.23
thermal expansion coefficient (at 20 to 300 °C) in 10^{-6} K^{-1}	3.25
T_g in °C	530
temperature in °C at $\lg\eta = 4$ (η in dPas)	1260

quality of the composite. On the other hand, it was stated [16] that the LMR is only valid when the elastic constants (Young's modulus and Poisson ratio) of fibres and matrix are equal; otherwise the effective thermal expansion coefficient is composed by portion of an elastic and a thermal extension. The elastic portion of extension is the consequence of the internal stress of the composite which is produced during the cooling from temperatures just above T_g of the matrix glass.

In a series of investigations [17] a hysteresis of the expansion curves was observed between heating up and cooling down which was explained by friction of the fibres at the interface fibre/matrix. This hysteresis effect is largest within the first temperature cycle and becomes smaller and smaller during the following cycles [13 and 18]. The hysteresis is larger the higher the maximum temperature [19] and it is larger for bidirectional than for unidirectional reinforcement [17]. After a storage time of 60 d the hysteresis effect of the composites was suppressed [13]. Also the hysteresis was suppressed after a modification of the hot-pressing conditions by which a stronger bonding was produced between fibre and matrix and the friction due to the relative movement along the interface fibre/matrix was prevented [20].

The expansion coefficient of carbon fibre/glass composites perpendicular to the fibre axis is by far larger than that parallel to it [13] due to the large anisotropy of this fibre type which corresponds in its crystalline modification to that of graphite. In the plane of the hexagonal graphite lattice the bonding force is very large (σ bonding), perpendicular to that the bonding is very weak. In carbon fibres the lattice planes are oriented parallel to the fibre axis [21].

The objective of this paper is to investigate the thermal expansion behaviour of various fibre-reinforced glasses in order to clarify the discussed discrepancies in literature and to complete in a systematic way the knowledge about the expansion behaviour of those composites which have given optimum properties so far. Reinforcement fibres are SiC fibres and various types of commercially available carbon fibres with different properties and preparation treatments. The glass matrix is DURAN glass from Schott Glaswerke, Mainz (Germany). The priority of the experiments will be the determination of the longitudinal expansion coefficients for the unidirectional reinforcement but also the transversal expansion coefficients will be regarded and the longitudinal expansion coefficients for bidirectionally reinforced composites.

2. Experimental

The composites were prepared by the sol-gel-slurry method. Details about this method are given in various papers [8 to 10]. In order to reinforce the matrix glass DURAN various hs(high-strength)- and hm(high-modulus)-carbon fibres from Toray (Toray-Industrie Deutschland GmbH, Frankfurt/M. (Germany)) and the SiC fibre Nicalon NL202 (Nippon-Carbon, Tokyo (Japan)) were used. Table 1 contains characteristic properties of the fibres and table 2 those of DURAN glass.

The thermal expansion coefficients are obtained from a dilatometer of Bähr Thermoanalyse GmbH, Hüllhorst (Germany), with a precision of $\pm 1 \cdot 10^{-7}$ K^{-1}. This dilatometer works by means of the difference principle with the help of a reference sample (silica glass rod ($50 \times 4 \times 4$) mm^3) of well-known expansion behaviour and with an inductive differential transformer. The samples have the same dimensions as the silica glass rod. This principle has the advantage that possible errors of the apparatus are eliminated. The surrounding space of the sample was permanently protected by argon gas flow to prevent oxidation of the fibres. The error of the temperature measurement is ±2 K.

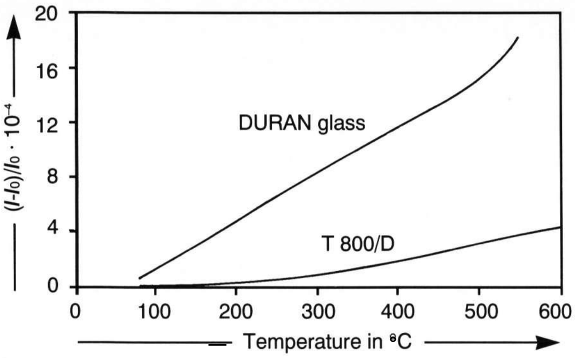

Figure 1. Thermal expansion curve of a unidirectional composite carbon fibre T800/DURAN glass (V_f = 40 vol.%) compared to that of unreinforced DURAN glass. (Here and in the following figures the letter "D" always stands for "DURAN glass" as the designation of the composites.)

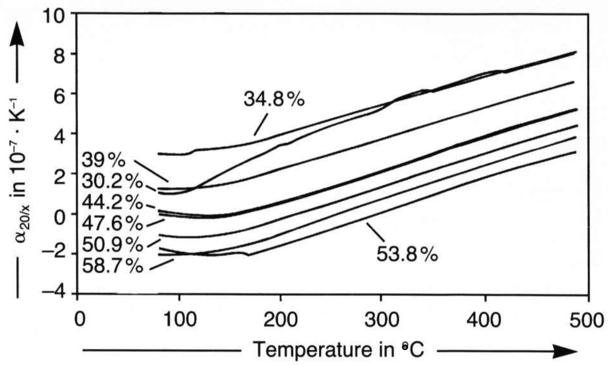

Figure 2. Mean longitudinal expansion coefficient $\alpha_{20/x}$ versus temperature of composites hs-carbon fibre T800/DURAN glass at various fibre vol.% concentrations.

3. Results

The results of the dilatometric measurements are given in diagrams of the mean expansion coefficient with respect to temperature intervals from room temperature up to a temperature which can be read from the abszissa of the plots, $\alpha_{20/x}$. In some cases the thermal alteration in length is drawn during definite cooling rate of the samples in order to compare this with that of the heating-up curve. With respect to the accuracy of the measurements ($\pm 1 \cdot 10^{-7}$ K^{-1}) the here investigated composites did not show any hysteresis even not for freshly prepared composites. These experiments were made usually up to 500 °C which is slightly lower than T_g of the matrix glass (530 °C). But also no hysteresis was observed when the samples were heated up to 580 °C. In these case the well-known bend of the transition branch of glass could not be detected (figure 1) because of the dominant influence of the fibre expansion behaviour.

The effect of the temperature on the longitudinal expansion of carbon fibre/DURAN-glass composites is seen from figure 1 as compared to the unreinforced DURAN glass. Reinforcement fibre is here the hs-carbon fibre Toray 800 with a content of 40 vol.%. The incorporation of the carbon fibres into the DURAN glass matrix leads to a drastic reduction of the longitudinal expansion. For this reason also the glass transition effect at 530 °C is not detectable any more after fibre reinforcement.

Figure 2 shows the influence of the fibre content on the longitudinal expansion coefficient. With increasing fibre concentration the expansion coefficient of the composites becomes smaller as expected because the portion of that phase with the very small expansion coefficient and with the large Young's modulus is increased.

The longitudinal expansion coefficients of various composites versus temperature are plotted in figure 3. All composites have the same fibre concentration of 40 ± 2 vol.%. The larger the Young's modulus of the fibre type is, the lower is the thermal expansion coefficient of the composite. This behaviour is caused by two parallel acting effects; first, the larger Young's modulus of the fibre leads to an increasing dominance on the expansion of the whole composite, second, the hm-carbon fibres have a better crystalline long-range order than those with a low Young's modulus [21] and as a consequence of that the expansion coefficient of the hm-fibre in axial direction approaches that of graphite in hexagonal plane and becomes therefore smaller. In this context it should be pointed out that no exact values in literature exist on the expansion behaviour of the various carbon fibre types, a circumstance

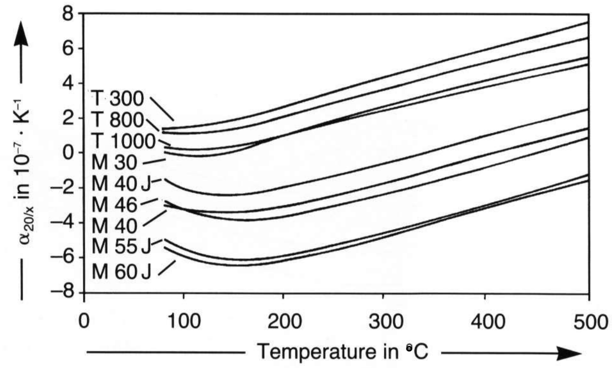

Figure 3. Mean longitudinal expansion coefficient $\alpha_{20/x}$ versus temperature of composites hs- and hm-carbon fibres/DURAN glass (V_f = 40 vol.%).

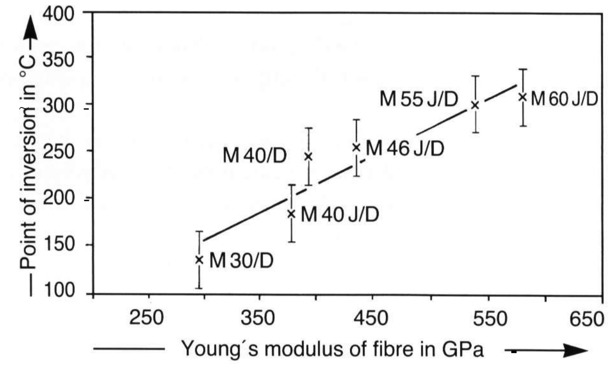

Figure 4. Point of inversion (contraction-expansion) versus Young's modulus of composites carbon fibres/DURAN glass (V_f = 40 vol.%) of the hm-fibre types.

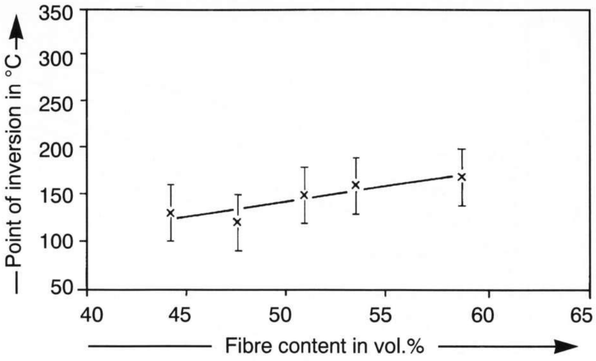

Figure 5. Point of inversion (contraction-expansion) of hs-carbon-fibre T800/DURAN-glass composites versus fibre volume content.

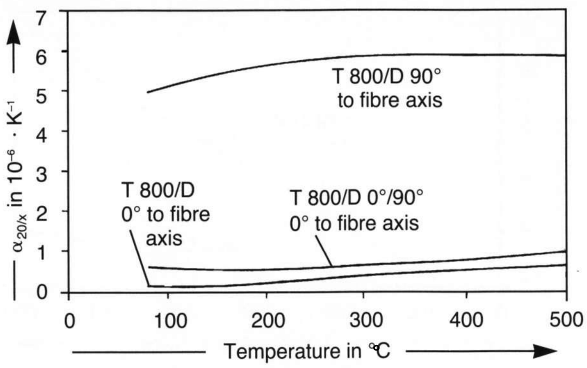

Figure 6. Mean expansion coefficient of unidirectional hs-carbon-fibre T800/DURAN-glass composites parallel and perpendicular to the fibre axis as compared with that of a 0°/90° ply composite (V_f = 40 vol.%).

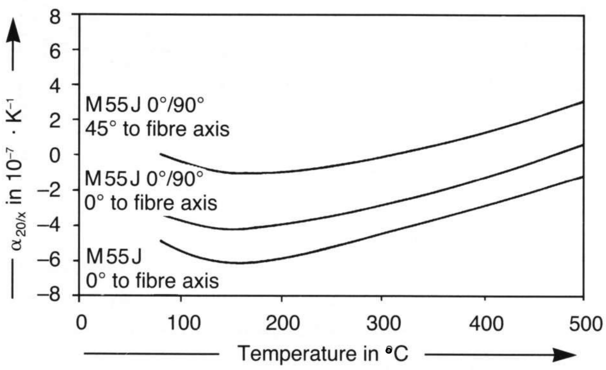

Figure 7. Mean expansion coefficient versus temperature for unidirectional hm-carbon-fibre M55J/DURAN-glass composites as compared with that of 0°/90° ply composites in 0^8 and 45° direction relative to the fibre axes (V_f = 40 vol.%).

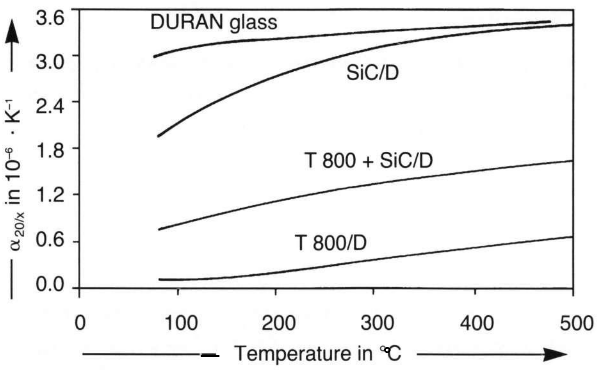

Figure 8. Comparison between the mean expansion coefficient of unreinforced DURAN glass versus temperature and those of DURAN glass composites with unidirectional SiC-, carbon T800- and 50:50 vol.% SiC-carbon T800 fibres (V_f = 40 vol.%).

which not at least may be due to the difficulty of an exact measurement of a material with such a small expansion coefficient.

While the hs-fibres produce a contraction and then an expansion of the composites only at fibre concentrations above 40 vol.%, the composites of the hm-fibres show the point of inversion from contraction to expansion already at lower fibre concentrations. The shift of this point with increasing Young's modulus of the fibres is seen from figure 4.

Figure 5 shows the point of inversion for the hs-carbon-fibre T800/DURAN-glass composites versus fibre volume content. The point of inversion is that temperature at which the dilatometer curve of a composite changes from a negative slope (contraction) to a positive slope (expansion) at which $\mathrm{d}L/(L_0\,\mathrm{d}T) = 0$ (or: $a_{\text{differential}} \equiv 0$).

In figure 6 the following expansion coefficients are compared: the longitudinal and transversal expansion coefficients of the hs-carbon-fibre T800/DURAN-glass composites and those of the 0°/90° ply composite with one fibre layer oriented to the direction of expansion measurement. The fibre content is 40 vol.% for all composites. The thermal expansion of the unidirectional composites perpendicular to the fibre direction is much larger than parallel to it. The expansion behaviour of the bidirectional reinforced composites is mainly determined by the 0° layer and the expansion coefficient is comparable to that of a unidirectional composite with a fibre content of 20 vol.% (compare the trend in figure 2).

The thermal expansion behaviour of the bidirectional reinforced hm-fibre-M55J/DURAN-glass composites as compared with the unidirectionally reinforced composites of the same fibre/glass combination is very similar (figure 7) to that described in figure 6. Even when the two fibre layers are oriented by an angle of 45° to the measurement direction, the expansion of the composites is only slightly larger than in axial direction.

Figure 8 gives a comparison of the coefficients of thermal expansion between unidirectionally SiC- and carbon-T800-fibre-reinforced DURAN-glass composites on the one hand and mixed carbon-T800/SiC-fibre hybride composites. In the latter composites both types of fibres are incorporated in equal volume concentrations. Also included in figure 8 is the expansion coefficient of the unreinforced DURAN glass. The composites with SiC fibres show significant larger expansion coefficients than those with carbon fibres. The expansion of the SiC fibres com-

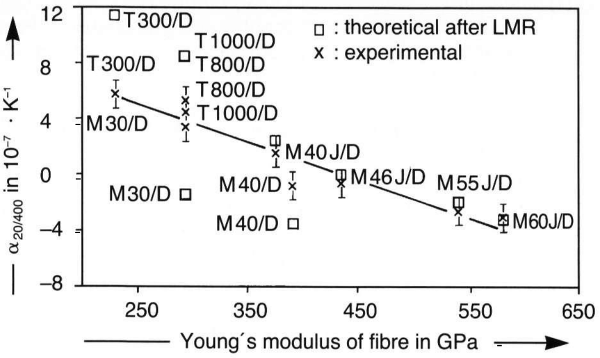

Figure 9. Mean expansion coefficient $\alpha_{20/400}$ of various carbon-fibre/DURAN-glass composites versus the Young's moduli of the reinforcing carbon fibre types V_f = 40 vol.% as compared with that calculated by the LMR (solid line).

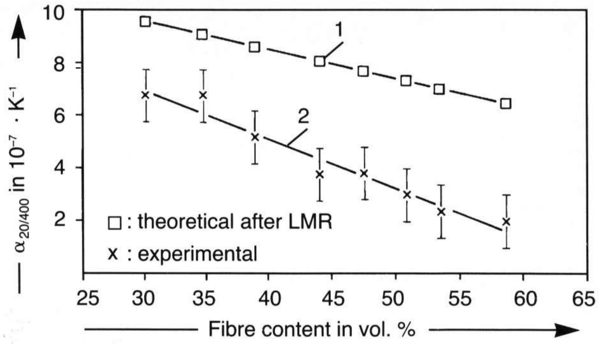

Figure 10. Mean expansion coefficient $\alpha_{20/400}$ of hs-carbon-fibre T800/DURAN-glass composites versus fibre volume concentration. Comparison between LMR calculation (curve 1) and experiment (curve 2).

posites are comparable with that of the unreinforced DURAN glass. In the SiC/carbon-hybride fibre composites the carbon fibres are dominant.

4. Discussion

Of the special interest is at first the question whether there is agreement between the measured thermal expansion coefficients and the calculated values by the LMR. The values of table 1 were used for the calculation, E_{comp} from the LMR, too.

Figure 9 demonstrates a very good agreement between theory and experiment in the temperature range 20 to 400 °C only for composites with fibres of the MJ series (see table 1). Larger deviations are found for composites with fibres of the T series (= hs-fibres) for which the experimentally determined thermal expansion coefficients are smaller than the calculated ones. The cause of this deviation may be seen in relatively unexact producer's data of the longitudinal fibre expansion coefficients, e.g. no temperature interval is given. But it is also possible that the expansion coefficient of the fibres of the T series is altered during the hot-pressing procedure by the release of nitrogen because hs-fibres are produced at relatively low temperatures and therefore they contain about 4 to 7 wt.% nitrogen [22] which is released at least partly at pressing temperatures of the composites. Those composites with fibres of the M series show larger experimentally determined expansion coefficients than theoretically calculated. Probably the producer's data are incorrect (for fibre type M30 no data were available, therefore, the value for fibre M40 was applied). For the fibre type M55J a mean expansion coefficient of $\alpha_{20/400} = -0.8 \cdot 10^{-6}\,\text{K}^{-1}$ was used. This is the mean value between that of the fibre type M46J which has a lower Young's modulus than the M55J type and that of the M60J type whose modulus is somewhat larger (see table 1).

Using the LMR for the calculation of the thermal expansion coefficient of the fibre M40, one obtains $\alpha_{20/400} = -0.84 \cdot 10^{-6}\,\text{K}^{-1}$. For the fibre type M30 the value $\alpha_{20/400} = -0.54 \cdot 10^{-6}\,\text{K}^{-1}$ is obtained in the same manner from the LMR.

Figure 10 shows the mean expansion coefficients for composites of the system carbon fibre T800/DURAN glass versus fibre volume content within a temperature range of 20 to 400 °C as compared to the theoretically calculated ones after the LMR. The theoretical values are systematically larger than the experimental values showing a similar slope. This indicates that the LMR seems to be suitable to describe qualitatively the longitudinal thermal expansion of unidirectional fibre/glass composites; quantitatively only then, when the expansion coefficient of the fibre type is well-known.

Perpendicular to the fibre axis the measured expansion coefficient ($\alpha_{20/400} = 5.0 \cdot 10^{-6}\,\text{K}^{-1}$) is smaller than the calculated one ($\alpha_{20/400} = 9.61 \cdot 10^{-6}\,\text{K}^{-1}$) (see figure 6). For calculation a radial expansion coefficient $\alpha_{20/400} = 35.5 \cdot 10^{-6}\,\text{K}^{-1}$ and a Young's modulus of 15 GPa were used for the fibre (producer's data). This difference may be a confirmation for the hypothesis of radial strain in the fibre which is built up during cooling down from just above T_g, resulting in a lower expansion of the composites perpendicular to the fibres. There must be a strong cohesion between the fibre and the glass matrix for building up these radial strains. In the case of carbon fibres this cohesion is caused by the rough surface of the fibres, resulting in a strong mechanical keying.

Due to the fact that the composites contract first and expand at elevated temperatures, a certain temperature with respect to the room temperature can be determined at which the total expansion is zero. This "zero expansion temperature" is that temperature at which the mean expansion coefficient is zero; i.e. at which the dilatometer curve itself changes from negative to positive values: $\Delta L/L_0 = 0$, or: $\alpha_{20/x} = 0$. Therefore, it is related to room temperature, i.e. to 20 °C. Because this saddle point from contraction to expansion depends for a given matrix glass on the Young's modulus as well as on the fibre content of the fibre type (increasing dominance of the fibres on the expansion of the whole composite) composites with different "zero expansion temperatures" can be tailor-made for special purposes. This correlation is demonstrated in figures 11 and 12. It is interesting for certain technical ap-

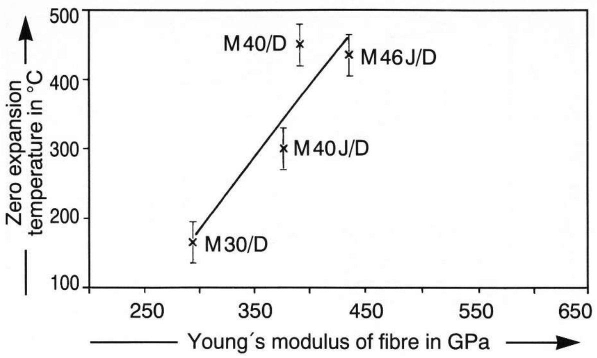

Figure 11. Zero expansion temperature of unidirectional DURAN glass composites with various hm-carbon-fibre types versus Young's modulus of the fibres (V_f = 40 vol.%).

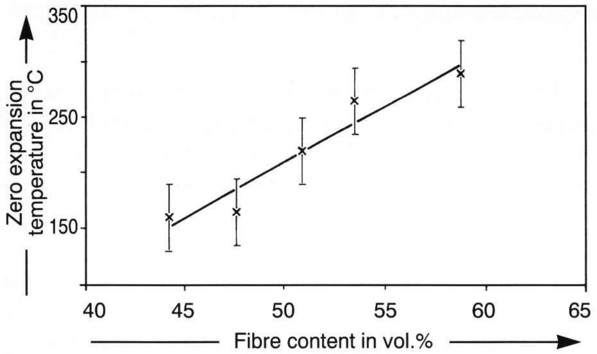

Figure 12. Zero expansion temperature of hs-carbon-T800 fibre/DURAN-glass composites versus fibre volume concentration.

plications to produce a "zero expansion composite" for a definite temperature interval. This can be done and controlled exactly for a zero expansion temperature by the choice of the fibre type and of the fibre volume concentration.

In total, the expansion coefficients of the carbon-fibre/DURAN-glass composites are very low also over a relatively large temperature range, usually lower than that of silica glass. It is positive that the thermal expansion of the investigated composites is free of hysteresis within the scope of accuracy of the measurements (see section 2.). This means that the composites are form-stable and do not show any permanent change in length with regard to their original lengths after one or more thermal cycles.

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