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SiC-fibre reinforced glasses — electrical properties and their application

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Abstract

Silicon carbide fibre reinforced glasses were investigated to create smart materials. The fibres were used not only as reinforcement but also as electrical sensors to detect the degree of damage or the temperature change of the specimen. Investigations showed that the electrical properties of the composite are not only influenced by the inlaid fibres but first of all by the in-situ formed carbon interface layer. Because of the electrical conductivity of the inlaid fibres and the fibre/matrix interface, the electrical properties of the composites have been used to detect the development of damage during mechanical testing. The development of damage due to fibre fracture and delamination under applied mechanical load in a unidirectionally reinforced specimen was monitored and a micro deformation analysis was performed simultaneously. Furthermore, local changes in temperature could be detected. Several measurements were taken to localize mechanical damage and temperature.

Keywords: Composites; Electrical conductivity; Glass-matrix; SiC-fibres; Smart materials

1. Introduction

In recent years, the role of online monitoring in the field of materials science has become more and more important, especially in safety-sensitive sectors like aerospace, where composites play an important role. It would be useful to have methods for accurate and reliable self diagnosis in the materials to facilitate fracture prediction.

Often composites consist of materials with large difference in properties, such as electrical resistivity and elastic modulus. Examples are composites containing fibres as a reinforcement. Some highly sophisticated structures of these composites make it possible to produce novel functions and "intelligent" mechanisms like self diagnosis in the materials. Such methods, which measure steadily and non-destructively the strength of a component could be realised by the introduction of special optical sensor fibres[1] or the implantation of whole smart composites as sensors in other materials.[2] However, problems of mismatching and stress introduction are associated with these methods. On the other hand, in some uses, already existing reinforcing elements could operate as sensors. Among these methods, there are already some by which electrical quantities were measured to obtain information about the "health" of a composite.[3–5] A requirement for this kind of smart composite is a large difference in electrical properties between matrix and reinforcing fibres. Preferably, the matrix is insulating and the fibres are electrically conductive. Often these so-called "smart" composites consist of carbon fibre reinforced polymers and are used for real-time non-destructive evaluation of delamination and damage control during tensile and fatigue testing.[6–14] But also fibre reinforced glasses and glass-ceramics could be used as a self detecting composite to check the degree of damage due to mechanical stress.[15]

In our study, a previously known system was investigated regarding its electrical properties. The reinforcing fibres were used as sensors, detecting electrical resistance as an indicator of mechanical strength, to create a fracture prediction technique. Furthermore, the appearance of a warm-up due to external heating during the application of a component should be detected, too.

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That means silicon carbide fibre reinforced glasses were investigated regarding their ability to inform about the degree of impairment after and during mechanical stress or about the component's present temperature.

2. Materials and experimental procedure

2.1. Sample preparation

SiC-fibre reinforced glasses were used for our investigations. Two types of specimens were chosen. At first, simplified models of SiC-fibre reinforced glasses were produced at Freiberg University (for fibre content see Table 2). The glasses DURAN® (borosilicate glass) and SUPREMAX® (alumosilicate glass) from Schott Glas (Germany) were selected and Tyranno TM-D1E08PX SiC-fibres from UBE Industries Ltd. (Japan) were chosen as reinforcing material. Later commercial specimens known as FORTADUR® from Schott Glas (Germany) were used. The commercial specimens were made of the glasses DURAN® and 8252 (alkali-earth alumosilicate glass) from Schott Glas (Germany) and of Tyranno TM-S1C16PX SiC-fibres from UBE Industries Ltd. (Japan). (Table 1, Fig. 1).

The preparation of all composites was made by a known slurry infiltration process described in detail elsewhere.[16,17] The fibre bundles were infiltrated with a slurry consisting of the glass powder and an organic binder. The prepregs were dried before densification. The densification was performed by a hot-pressing process under an inert atmosphere. In case of the specimens produced at Freiberg University, the hot-pressing temperature was 1100°C.

2.2. Measurements of electrical resistance vs mechanical load as well as heat treatment

Afterwards the reinforced glasses were cut into rectangularly shaped pieces. One part of the specimens was used to apply a three-point bending load or a pressure load on the samples while simultaneously the electrical resistance was measured. The other part of the specimens was heat treated while the electrical resistance was measured.

Mechanical testing took place at room temperature using a universal testing machine (INSTRON Int. Ltd, USA) while thermal treatments were performed in an alumina tube furnace in air or later for local resolution

Table 1
Properties and composition of glass matrices and SiC-fibres

	Glass matrix			SiC-fibre	
	Supremax®	Duran®	8252	TM-D1E08PX	TM-S1C16PX
ρ (g/cm^3)	2.57	2.22	2.63	2.37	2.45
$\alpha_{20/300}$ (10^{-6}/K)	4.1	3.3	4.6	3.1	3.1
T_g (°C)	730	525	725	–	–
Young's modulus (GPa)	87	63	81	182	187
Electrical resistivity (Ω cm)	–	–	–	1.9×10^3	17
Composition (wt.%)	SiO_2 52	SiO_2 79.7	SiO_2 59.9	Si∼52	
	Al_2O_3 22	B_2O_3 10.3	Al_2O_3 13.8	C∼32	
	P_2O_3 8	Na_2O 5.2	CaO 10.2	O_2∼12	
	MgO 7.5	Al_2O_3 3.1	BaO 8.8	Ti∼4	
	CaO 7	MgO 0.9	B_2O_3 4.5		
	B_2O_3 2	CaO 0.8	MgO 2.4		
	BaO 1.5		other 0.4		

Table 2
Specimens and their application

Matrix	SiC-fibre	Fibre volume fraction	Produced at	Reinforcing direction	Application	Reported in
SUPREMAX®	TM-D1E08PX	4.5±1%	Freiberg University	Unidirectional	3-point bending test	27, 28
DURAN®	TM-D1E08PX	4.5±1%	Freiberg University	Unidirectional	Thermal treatment	31, this report
DURAN®	TM-D1E08PX	see Fig. 2d	Freiberg University	Two-dimensional (perpendicular 0°–90°)	Thermal treatment, local resolution	29, this report
8252	TM-S1C16PX	40±5%	Schott Glas	Unidirectional	3-point bending test	29
DURAN®	TM-S1C16PX	40±5%	Schott Glas	Undirectional	3-point bending test, local resolution	30, this report
DURAN®	TM-S1C16PX	40±5%	Schott Glas	Unidirectional	Pressure load	this report
8252	TM-S1C16PX	40±5%	Schott Glas	Unidirectional	Thermal treatment	this report

by using a flame and heating the specimens directly at specific positions.

To detect the electrical resistance of the composites the specimens were connected to copper electrodes. Silver paint was used for electrical contacts. The type of connection depended on the kind of investigation, as shown in Fig. 2. A Keithley 2000 digital multimeter was used for the electrical measurements.

2.3. Optical measurements vs mechanical deformation

Furthermore, a new method of a micro deformation analysis, the so-called "grey scale correlation analysis", was performed with help of the Chemnitzer Werkstoffmechanik GmbH. This method is based on imaging of specimens during their deformation. A series of pictures is recorded in the course of specimen deformation and the positions of identical grey values in the images of different states of deformation — representing identical points of the specimen — are correlated to give the vectors of movement.[18,19] In our case, photos during the mechanical testing of the specimens were taken. These photos were digitized and the grey scale pattern of digitized photos of different stress situations were correlated. As a result, an optical impression of the deformation of the specimen as well as figures of the displacement as vector diagrams were obtained. From these diagrams, bending lines could be calculated.

A summary of the specimens used and their experimental application is given in Table 2.

3. Results and discussion

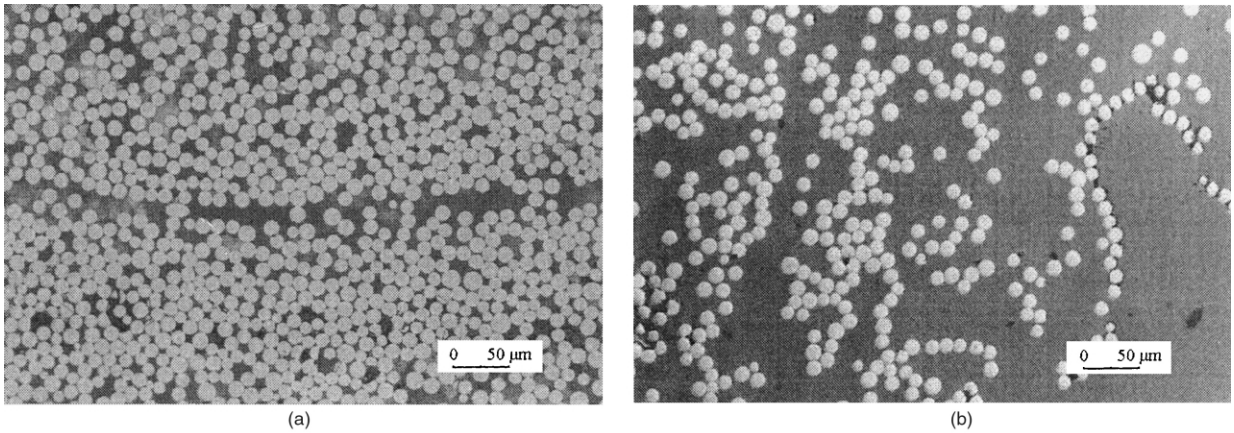
3.1. Structural studies

A fundamental feature of SiC-fibre reinforced glasses is the large difference in electrical properties between fibres and matrix. Because the glass is an insulating matrix, an assumption was made at first that the semiconducting SiC-fibres could affect the electrical properties of the composite. However, the appearance of a carbon layer at the fibre-matrix interface during hot-pressing was found to have a much larger influence on the composites' electrical resistivity.

The in-situ formation of such a carbon interface has been widely observed in glass and glass-ceramic matrix composites reinforced with SiC-fibres and has been reported by several authors.[20–24] Usually the carbon interface between the fibre and the matrix provides a weak line and is responsible for the high toughness of these composites because it allows crack deflection, interfacial debonding and fibre pull-out. Such phenomena are of fundamental importance for the reinforcement of composites with brittle matrices.[25]

A scheme of such a carbon layer is given in Fig. 3. It shows the difference in structure between the existing carbon interface (turbostratic carbon) and a carbon layer perfectly structured (graphite). The two-dimensional honeycomb structure of turbostratic carbon consists of uncorrelated individual carbon layers. Weak disorder results in stacking faults giving rise to a small increase in the interlayer distance. Therefore, the stacking of the individual carbon layers becomes uncorrelated and turbostratic carbon occurs. The electronic structure of turbostratic graphite, a zero gap semiconductor, is qualitatively different from that of ideal graphite, a semimetal with a small band overlap.[26]

This carbon interface affects the electrical properties of the SiC-fibre/glass-matrix composites. This assumption was supported by the values determined for the electrical resistivity of the single fibres as compared to the composites. The used SiC-fibres were stable in their mechanical and electrical properties up to about 1200°C.[27,28] Table 3 shows the measured values of electrical resistivities of the SiC-fibres and of the composite.



(a) (b)

Fig. 1. Examples of cross sections of investigated specimens. (a) SiC-fibre reinforced DURAN®-glass matrix produced by Schott Glas (Germany). (b) SiC-fibre reinforced DURAN®-glass matrix produced at Freiberg University.

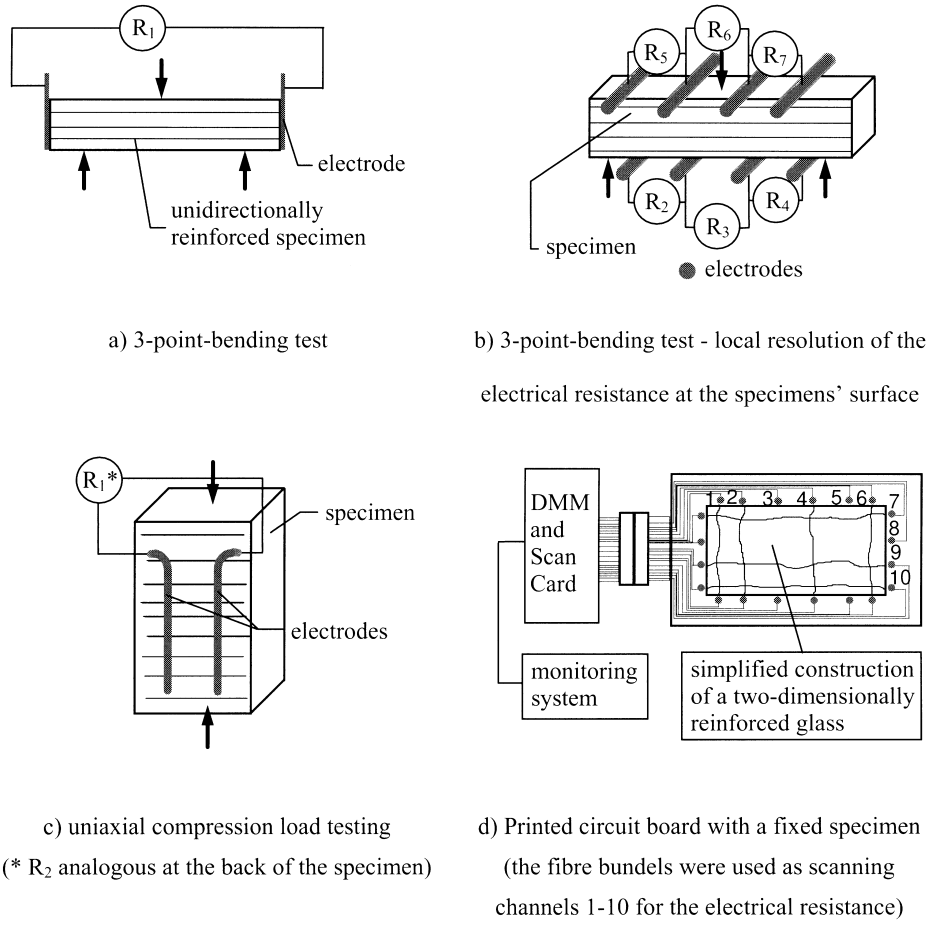


Fig. 2. Models of the measuring principles. (a) 3-point-bending test. (b) 3-point-bending test — local resolution of the electrical resistance at the specimens' surface. (c) Uniaxial compression load testing (*R_2 analogous at the back of the specimen). (d) Printed circuit board with a fixed specimen (the fibre bundels were used as scanning channels 1–10 for the electrical resistance).

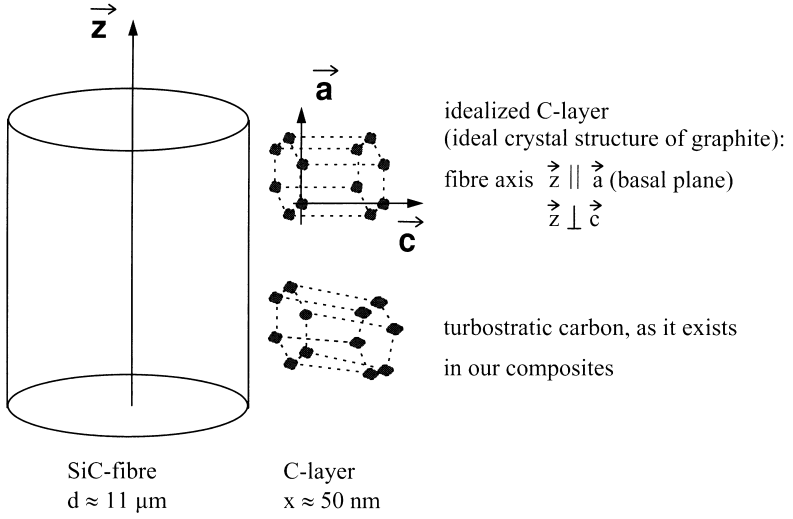


Fig. 3. Scheme of the carbon layer at the fibre–matrix interface after hot-pressing.

The electrical resistivity of the composite is much lower than that of the SiC-fibres itself. Consequently there must be something else in the composite which is electrically conductive.

Accordingly we assumed a carbon layer of a thickness of about 50 nm to explain the observed decrease of the electrical resistance of the composite after hot pressing. This estimation is in agreement with data from the literature.[20–24] Comparing the measured results with the calculated value of the electrical resistivity of the carbon layer at the fibre–matrix interface (Table 3), it is clearly seen that the electrical properties of the composite are affected by the carbon interface.

It should be noted that in our case, the type of glass matrix used here has no influence on the electrical resistivity of the composites. However, an increase in the isotropy of the fibres' architecture in the composite, from unidirectional via perpendicular to random orientated, leads to an increase in the electrical resistivity of the whole composite as we have published elsewhere.[29]

3.2. Mechanical testing

In case of specimens on which mechanical load was applied, it was possible to monitor the degree of impairment during mechanical stress based on changes in electrical resistance, because a change of the load always leads to a change in electrical resistance. At first, the unidirectionally reinforced specimens were investigated measuring the electrical resistance parallel to the reinforcing direction by applying a three-point bending load (Fig. 2a). A distinction could be made between fibre fractures and delamination connected with fibre pull-out before the final macroscopic failure of the component occurred.[27,28] Fig. 4 shows typical results for this kind of measurement. It is clear to be seen that the bend points of the resistance curve were connected to fibre fractures. In this case the load appearing on the specimen drops dramatically. On the other hand a steady rise in resistance is connected with delamination and fibre pull-out.

However, the behavior of the electrical resistance before a *first* matrix crack occurred is much more important with regard to fracture prediction, since it should arise the occasion to give warning before cracking starts.

Fig. 5 shows this first part, until a first matrix crack occurred, of such a relationship between applied load, deflection and electrical resistance to unidirectionally reinforced commercial composites. The value for the electrical resistance was normalized to show more clearly its variation during mechanical testing. Therefore, the initial value of the electrical resistance was 100%. From Fig. 5 it can be seen that at the beginning of the mechanical loading, the electrical resistance increased with increasing load. Though the change in electrical resistance seemed to be not very significant, it was observed in all the cases. By using a new method of a micro deformation analysis described above we established that this first increase in resistance is just influenced by destroying the fibre/matrix interface, consisting of the electrically conductive carbon layer, and not by matrix cracking with fibre pull-out and fibre

Table 3
Comparison of electrical resistivities

Material	Measuring direction	Electrical resistivity (Ω cm)
SiC-fibre	Parallel fibre axis	1.9×10^{3}
SiC-fibre/glass composite	Parallel fibre axis	$\leqslant10^{-3}$
C-layer at the fibre-matrix interface (assumptions: 50 nm thick, parallel connection of resistances)	Parallel fibre axis	2.7×10^{-3}

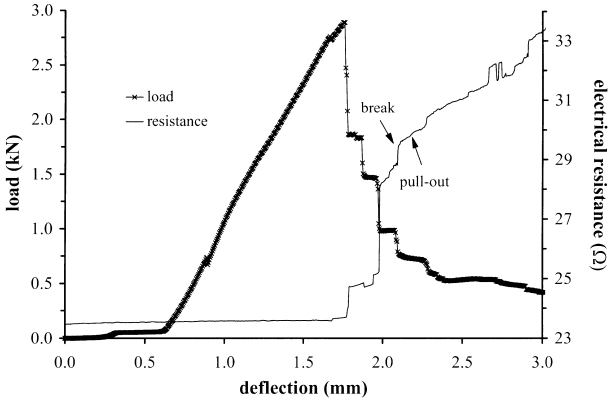


Fig. 4. Load–deflection curve and electrical resistance when applying 3-point bending load to a specimen from Schott Glas (Germany), cf. Fig. 2a for the measuring principle.

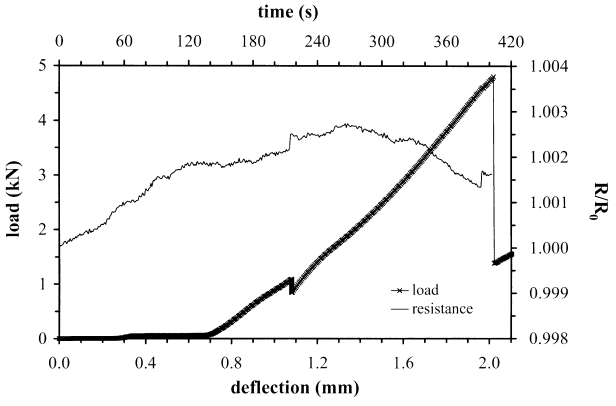


Fig. 5. Load–deflection curve and electrical resistance when applying 3-point bending load to a specimen from Schott Glas (Germany) before a first matrix crack occurred, the initial value of the electrical resistance was determined to be 100%, cf. Fig. 2a for the measuring principle.

breaking.[30] In Fig. 6, a vector diagram and the calculated bending lines for a particular situation during the mechanical testing is shown. The bending lines were calculated from the figures of displacement corresponding to a certain state of stress in comparison to the specimens' situation at the beginning of the measurement (no stress and no deformation of the specimen). The bending lines give an impression how the specimen is deformed in its regions of different stress situations after introducing a certain state of stress, corresponding to a certain experimental time of stress increase.

The results of this micro deformation analysis support our position. The correlation of the grey scale pattern showed no deformation until the resistance curve reached its maximum and no evidence of a matrix cracking during this time was found. Afterwards the destruction of the matrix started (Fig. 7, first deformation after 258 s). So the subsequent decrease of the electrical resistance (Fig. 5) could be as a result of a rejoining of some already destroyed parts of the composite under pressure or it could be due to a change in alignment of the turbostratic carbon into a straightened more uniform C-layer (Fig. 3). A more uniform alignment of the carbon layer would improve the electrical conductivity and consequently the electrical resistance would decrease.

To confirm this assumption, we reconfigured the measurement to obtain a local resolution of the load condition of the specimens. Additional small electrodes were fixed at certain points of the specimens surface (Fig. 2b) and changes in the electrical resistance of particular locations close to the surface and parallel to the reinforcing direction of the specimen were established independently from each other (Fig. 8). It was thus possible to detect localized damage of the composite by measuring the array of electrical resistances. It is clearly seen that in the region of compressive stress (top of the specimen) the resistance decreases while in the region of tensile stress (bottom of the specimen) the resistance slightly increases because of destroying the carbon-rich fibre/matrix interface, or later, because of fibre breaking

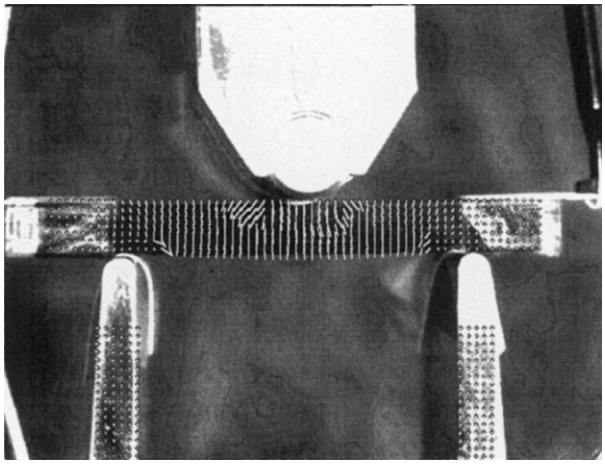


Fig. 6. Results of the micro deformation analysis (after 258 s of stress increase): bending lines calculated from with the vector diagram of the gray scale pattern above for the three stress regions (top, centre, bottom) of the specimen in the investigated situation at Fig. 5.

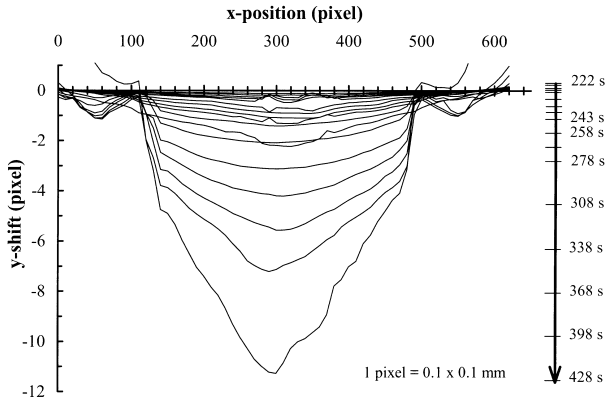
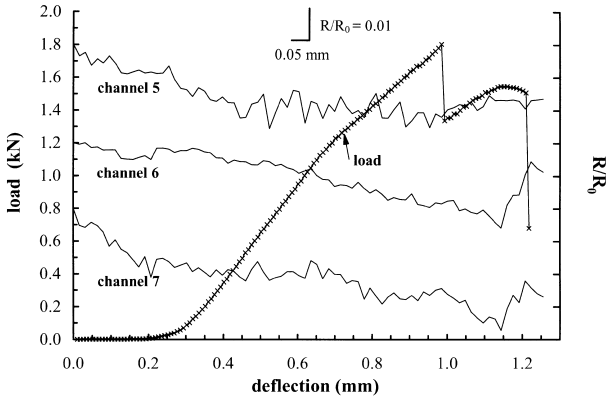
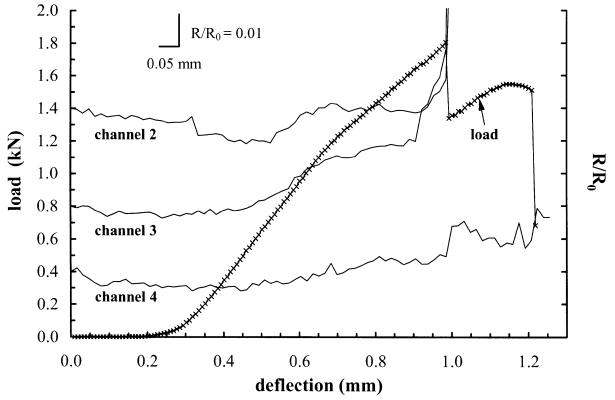


Fig. 7. Combination of all bending lines calculated for the complete 3-point-bending test (bottom of the specimen in Fig. 5 and 6, region of tensile stress), each bending line corresponds to a deformation after a certain time of stress increase.



a) compressive stress



b) tensile stress

Fig. 8. Load–deflection curve and electrical resistance when applying 3-point bending load to a specimen from Schott Glas (Germany), cf. Fig. 2b for the measuring principle. (a) Compressive stress. (b) Tensile stress.

and delamination. The decrease of the resistance in the region of compressive stress resulted from compressive stress perpendicular to the reinforcing direction (piezo-resistivity). Since the contribution of the SiC-fibres to the conductivity is about two orders lower than that of the interface layer, also this decrease of resistance should arise from the carbon layer. Obviously, this compressive stress facilitates changes in the alignment of the turbostratic carbon into more uniform C-layers at the fibre/matrix interface leading to an improvement in conductivity. The tensile stress, however, evidently leads to crack introduction and consequently to an increase in the electrical resistance. Later the decrease in resistance is superposed by an increase due to fibre fracture and delamination caused by the stress situation during further testing.

The argument that compressive stress leads to a change in alignment of the C-layer at the fibre/matrix interface was confirmed by determining the electrical resistance during a pure uniaxial compressive load perpendicular to the fibres direction of the specimen. Fig. 9 shows that the electrical resistance decreases because of the compressive load. The electrical resistance at particular locations close to the surface of the specimen (cf. channels 5–7 in Fig. 8a) shows a sharp decline.

Furthermore, it was shown with the help of cyclically loading (3-point bending test and pressure load) that these changes in the electrical resistance were reversible until the first crack occurs.

3.3. Temperature influence

As a second part of the investigation, measurements due to changes in temperature and changes in the composites' structure due to thermal exposure of the specimens were carried out. First the SiC-fibres were examined regarding their temperature dependence of electrical resistance.[31] The SiC-fibres behaved as is typical for semiconducting materials up to temperatures of about 500°C. For higher temperatures it was found that there might be a change in the structure of the additional free carbon of the SiC-fibres, since the electrical resistance during cooling was slightly lower than that of the heating period. Apparently, the free carbon is forming a percolating network step by step.[31] Simultaneously, it became more graphite-like, as confirmed by X-ray diffraction curves.

The electrical properties of the composites were also determined.[32] Specimens hot-pressed at different temperatures and their electrical resistivities were investigated (Fig. 10). It is clearly seen that the electrical resistivity decreases with an increase in hot-pressing temperature. Since the original electrical resistivity of the fibres (Table 1) is much higher than the measured values at 800°C, the first decrease in electrical resistivity must be connected with the formation of a carbon network within the SiC-fibres, as described above, since the reaction forming a carbon interphase not yet takes place at this temperature. But the further descend (temperatures

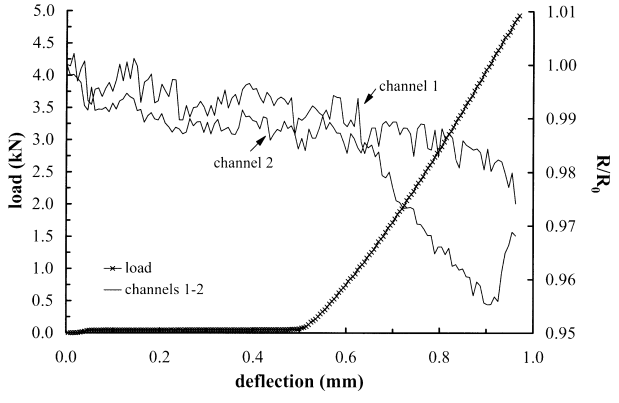


Fig. 9. Load–deflection curve and electrical resistance when applying uniaxial compression load to a specimen from Schott Glas (Germany), the initial value of the electrical resistance was determined to be 100%, cf. Fig. 2c for the measuring principle.

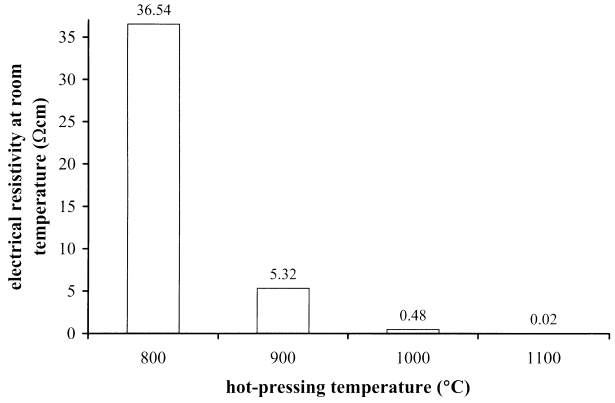


Fig. 10. Electrical resistivity after hot-pressing (specimens made at Freiberg University).

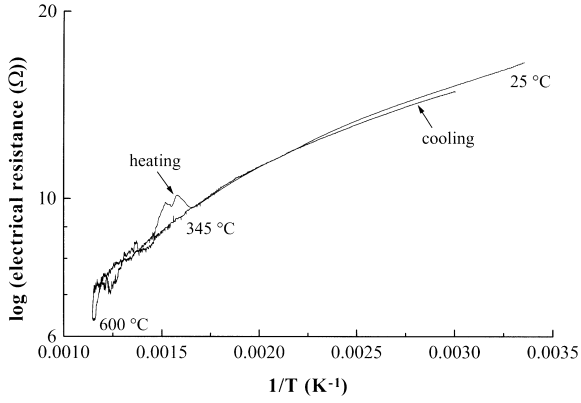


Fig. 11. Electrical resistance of a composite from Schott Glas (Germany) during heating to and cooling from 600°C.

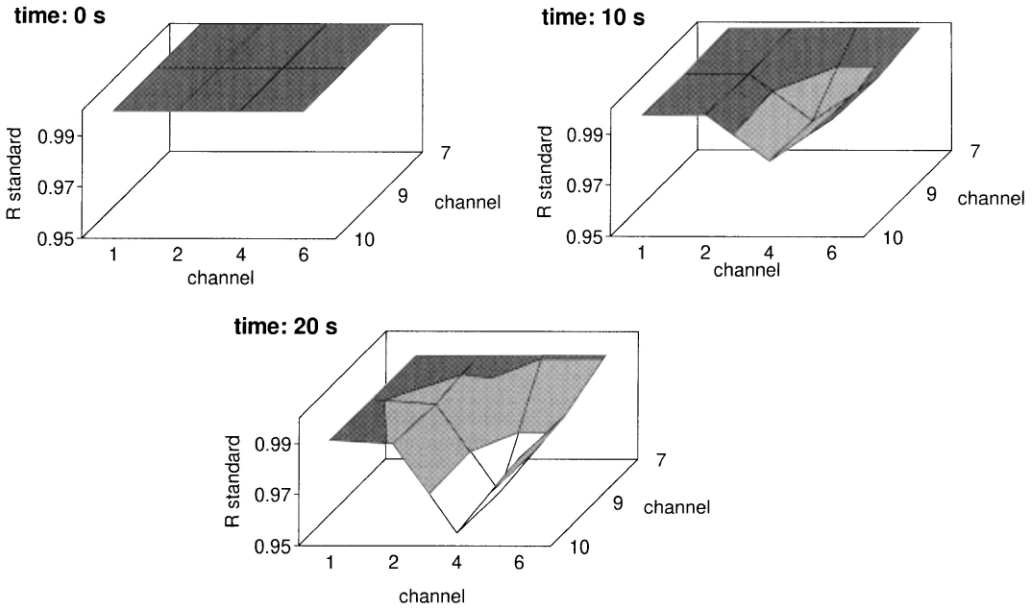


Fig. 12. In-situ detection and local resolution of the change in electrical resistance of a specimen due to locally increasing temperature (heating the specimen at channel 4, cf. Fig. 2d).

above 800°C) of electrical resistivity must be connected with the appearance of the carbon layer at the fibre/matrix interface, since the decrease with temperature is significantly stronger than that one of the fibres alone. A superposition of both effects could lead to the steep fall of the electrical resistivity.

The above investigations and observations allowed an in-situ detection of changes of the electrical resistance during heat treatment of the composites. SiC-fibre unidirectionally reinforced glasses were heated up from room temperature to 600°C and then cooled (Fig. 11). At first, it can be seen that the composites behave like semiconductors and that there is no hysteresis between heating and cooling period. In case of the electrical resistance at temperatures higher than 345°C the organic part of the contacting material started to burn out and the electrical resistance measured at this time started to become unstable. An in-situ detection of the electrical resistance at temperatures higher than 600°C was not possible due to lack of a suitable contacting material. However, up to 600°C no structural changes in the composite were apparent, based on our resistance measurements.

Simplified constructions of two-dimensionally reinforced specimens (Fig. 1d) were used to establish local resolution of changes in temperature as reported already.[29] Therefore, specimens were fixed on a printed circuit board and heated at certain positions by a flame. The heating led to a descent in electrical resistance and the online monitoring system showed this descent at the same time (Fig. 12). Because the system showed a very short response time, an application as an online monitoring system of temperature could be possible.

4. Conclusion

Silicon carbide fibre reinforced glasses have been investigated to create smart materials where the reinforcing fibres execute an additional function. The double function is possible since the fibres and the fibre–matrix interface with its carbon layer control the electrical conductivity of the composite. The fibres function not only as a reinforcement, but also as a sensor to detect damage of the composite due to mechanical load or to detect temperature differences.

Using unidirectionally reinforced specimens, a simultaneous detection of mechanical stress and electrical resistance has been carried out vis-à-vis.

(a) The measurement of the electrical resistance of the whole specimen at one position parallel to the reinforcing direction during a 3-point bending test demonstrated a correlation between electrical and mechanical properties.
(b) A micro deformation analysis by means of correlation analysis of grey scale pattern indicated that the first increase in resistance under load is influenced by a destruction in the fibre/matrix interface with the electrically conductive carbon layer.
(c) The detection of the electrical resistance at different positions of the specimens surface under a 3-point bending test and under pure compression load established the reason of the temporary decrease of the electrical resistance to be the change in conductibility of the carbon layer, influenced by a change in the alignment of the turbostratic carbon, obviously. Besides, a localization of

the damage showed the possibility of a final failure prediction.

The influence of temperature on the electrical properties of the SiC-fibres and the composites has been investigated. A simplified construction of two-dimensionally reinforced glasses was used to resolve changes in temperature and localization of the temperature influence was detected.

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