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Conductive SiC-fibre Reinforced Composites as a Model of 'Smart Components'

Beate Fankhänel,[a]* Eberhard Müller,[a] Torsten Fankhänel[b] and Winfried Siegel[b]

[a]Freiberg University of Mining and Technology, Institute of Ceramic Materials, Gustav-Zeuner-Straße 3, D-09596 Freiberg, Germany
[b]Freiberg University of Mining and Technology, Institute of Experimental Physics, Silbermannstraße 1, D-09596 Freiberg, Germany

Abstract

Electrical and mechanical properties of ceramic composites are investigated to develop 'smart components' and establish a fracture prediction technique by measuring a change of electrical resistance under applied mechanical load in electrically conductive composites. One part of those possible 'smart components' is based on silicon carbide fibre-reinforced composites. First the SiC-fibres were checked to determine if there exists a correlation between the electrical resistivity and the mechanical strength of SiC-fibres. Two ways were followed to produce the fibre-reinforced glass-matrix composites. It was found that both ways are appropriate to create dense samples. The following determination of the mechanical strength with in situ *detection of the damage degree showed that the strain and the development of damage due to fibre fracture and delamination can be detected. © 1998 Elsevier Science Limited. All rights reserved*

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1 Introduction

Ceramic composites are widely investigated in order to improve mechanical, electrical and/or thermal properties of the ceramic matrix material. Outstanding examples are ceramic composites containing fibres as a reinforcement. Often these composites consist of materials with large difference in properties, such as electrical resistivity and elastic modulus. Fabrication of such composites makes it possible to produce novel functions and 'intelligent' mechanisms such as self diagnosis[1–5] in the materials.

The idea of obtaining information about the structure of a composite by electrical measurements is not absolutely new. Methods already exist by which electrical quantities are measured to obtain information about the amount, distribution and orientation of the reinforcing materials. The aim of this study is to develop 'smart components', that are able to give information about their degree of impairment after and during mechanical or thermal stress.

Composites that consist of an insulating matrix made of borosilicate glass and electrically conductive SiC-fibres are fabricated. The SiC-fibres are not only used as a reinforcement but also as electrical sensors to detect the degree of damage or the temperature of the specimen, because of their electrical conductivity and temperature dependence. Thus they were checked to determine the relationships between their electrical resistivity, their tensile strength and thermal exposure.

2 Materials and experimental procedure

The alumo borosilicate glass Supremax® from Schott Glaswerke (Germany) and the borosilicate glass foil AF 45® from DESAG (Germany) were selected for the investigations. As reinforcing components two different types of Tyranno SiC-fibres from UBE Industries Ltd (Japan) were chosen for the study (Table 1).

The preparation of the unidirectionally reinforced composites was made by a sol–gel slurry method similar to processes described elsewhere.[6–9] The fibre bundles were infiltrated with a slurry consisting of the glass powder and a silica sol.

Afterwards the prepared prepregs were dried before densification. The densification was performed in a high temperature graphite furnace

*To whom correspondence should be addressed. Fax: 0049 0 3731 39 3662; e-mail: fankhaenel@anw.ikw.tu-freiberg.de

Table 1. Properties of the used glass matrices and SiC-fibres

	Glass matrix		*SiC-fibre*	
	Supremax® glass	*AF 45®*	*Tyranno 1*	*Tyranno 2*
ρ (g cm^{-3})	2·57	2·72	2·37	2·43
$\alpha_{20/300}$ (10^{-6} K)	4·1	4·5	3·1	3·1
T_g (°C)	730	662	—	—
Young's modulus (GPa)	87	66	182	175
Specific resistance (Ω cm)	—	—	$1{\cdot}9\times10^3$	$5{\cdot}1\times10^0$

under high gas pressure and an inert atmosphere, similar to a hot pressing process. The pressure of 8 MPa was held during 15 min at a maximum temperature of 1100°C.

A second method to prepare prepregs includes the application of thin glasses. Now the fibres are drawn through the silica sol in itself and paste with the glass foil. The thickness of the foils is declared to be 50 μm. Dense samples are prepared analogously to the densification process described above.

Parallel to these investigations the SiC-fibres were examined regarding their electrical properties. Several heat treatments took place in an alumina tube furnace under different conditions. Afterwards their electrical resistivity and tensile strength were determined.

The reinforced glasses were cut and rectangular shaped specimens were used for the 3-point bending tests at room temperature. The composites were contacted by electrodes on the fibre ends to detect electrical signals coming from the inlaid SiC-fibres. All mechanical properties were determined using a universal testing machine from Instron International Ltd (USA). The Scanning Electron Microscope DSM 960 from Zeiss (Germany) was used for the images in Figs 4 and 5

3 Results and discussion

The two ways to produce SiC-fibre reinforced composites are found to be suitable to obtain dense samples. In comparison to previous studies of other authors, using the same materials,[6–9] it was possible to decrease the hot pressing temperature by about 200 K to 1100°C as mentioned above. Using a temperature higher than 1100°C more porous samples were produced for one component of the Supremax® is P_2O_5. This oxide tends to evaporate and to create pores.

Figs 1 and 2 show a cross section of a specimen made of Supremax® and Tyranno 1 fibres before and after densification. It is clearly to be seen that the fibre bundles are well infiltrated. The same results were achieved for the composites made of glass foil. But it was necessary to reduce the gas pressure during the densification process to 3 MPa

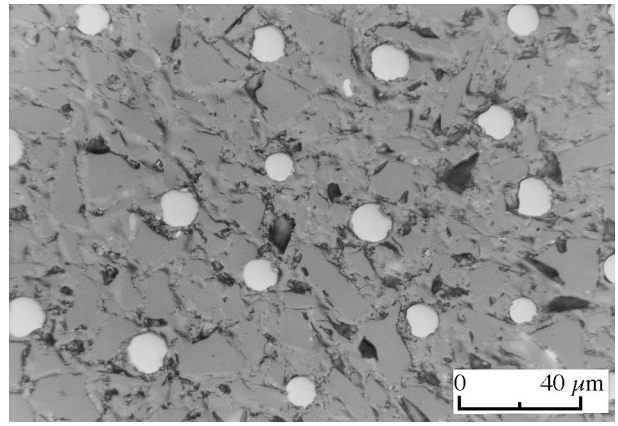

Fig. 1. Cross section of a specimen made of Supremax® and Tyranno 1 fibres dried after slurry infiltration.

because a higher pressure will push away the glass and separate it from the fibres. The chemical composition of the glass foil is slightly different from those of the Supremax® and the viscosity of the glass foil will be much lower than that of Supremax® at the same time of the densification process.

Figure 3 shows the results of the electrical resistivity measurements of the fibres with varying heat treatment temperatures for 1 h in an argon atmosphere. For Tyranno 1 the resistivity decreases gradually up to 1000°C and shows a dramatic fall of the order of 10^3 between 1200°C to 1600°C. In the case of Tyranno 2 the resistivity first decreases gradually up to 800°C but it

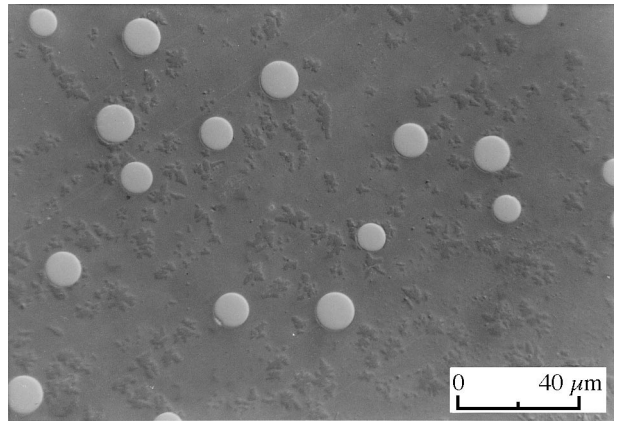

Fig. 2. Cross section of a specimen made of Supremax® and Tyranno 1 fibres by slurry infiltration after the densification process.

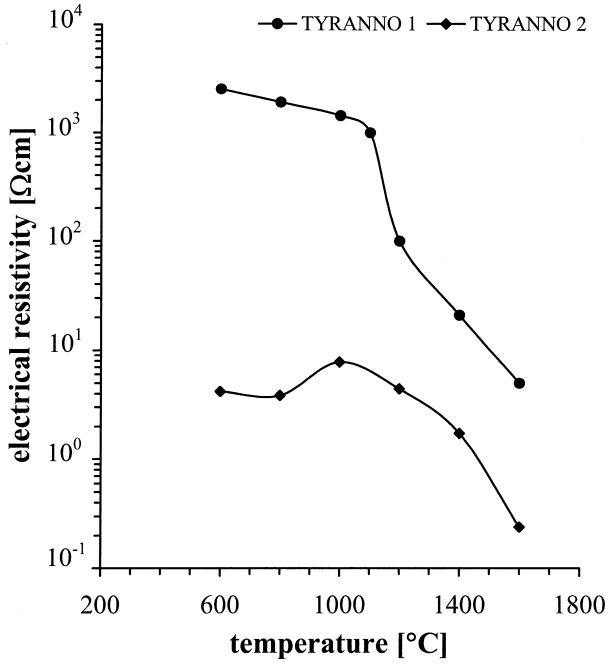

Fig. 3. Electrical resistivity of Tyranno fibres after 1 h heat treatment in an argon atmosphere.

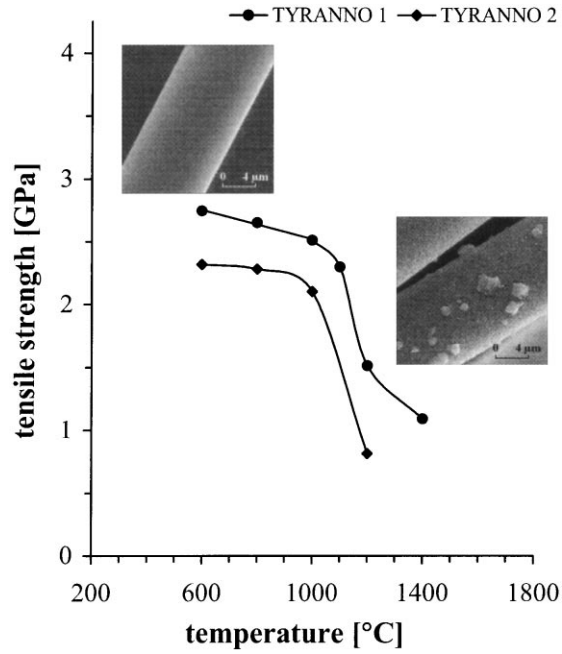

Fig. 4. Tensile strength of Tyranno fibres after 1 h heat treatment in an argon atmosphere (the pictures show Tyranno 2 fibres after a heat treatment of 600°C and of 1400°C).

shows a small increase up to 1000°C. Afterwards the resistivity decreases again. For comparable measurements a similar course was observed by Narisawa.[10]

Comparing these results with the results determined for the strength measurements (Fig. 4) a clear correlation between the fibres' conductivity and mechanical strength can be perceived. That means that if the fibres' strength decreases, their electrical resistivity decreases, too. Both effects can be seen as a consequence of a crystallization process. The statement is confirmed by additional examinations of the SiC-fibres by X-ray and scanning electron microscopy (Fig. 4). The diffraction peak of graphite like carbon became more distinctly and the broad SiC peak became sharper and an formation of oxide crystals on the fibre surface began. Thereby the starting temperature of rapid crystallization and grain growth under argon for Tyranno 2 was determined to be about 800°C. By way of contrast, the temperature for Tyranno 1 was determined to be about 1000°C. The differences between the two types of fibres lie in the higher oxygen content of Tyranno 2, particularly

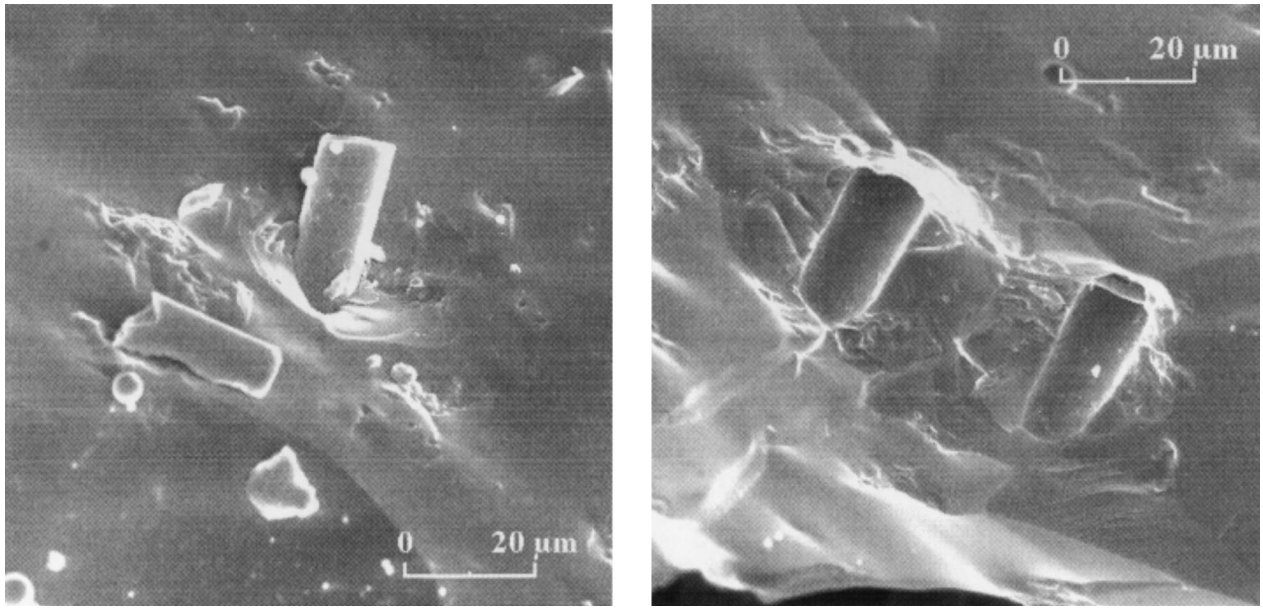

Fig. 5. Fracture surface of a specimen (corresponding to Fig. 2) after applying bending load.

in the different distribution of additional free carbon.

Other dependencies were investigated by the same way. In all cases, i.e. for other variables (e.g. atmosphere, time and heating velocity), we have found similar correlation between the fibres' conductivity and mechanical strength. That means if the fibres' strength decreases, dependent on the preliminary treatment and the damage degree, the electrical resistivity of the fibres decreases, too. But it is possible to say that the electrical properties of the fibres will always have defined values after the composites' production, dependent on the fibre's type.

However in all cases, that means for Tyranno 1 as well as for Tyranno 2, the electrical resistivity after the composites' production was determined to be 10^0 Ωcm. This is lower than one might expect it from Fig. 3. Such low values of the electrical resistivity are expected to occur after treatment at much higher temperatures. But the fibres showed no corresponding loss in their strength. The reason for this contradiction can be seen in the influence of the glass matrix during the composites' production. Hähnel for instance, found the appearance of a carbon layer on SiC-fibres in similar glass matrices after a hot pressing process.[11–13] Therefore it could be possible that carbon layers exist and that they are responsible for the measured low resistivity of the whole specimen. Another evidence that a carbon layer exists is the occurrence of fibre pull-out (Fig. 5).

Figure 6 shows the relation between applied load, deflection and electrical resistance by applying a three-point bending load to the composites. It is clear that the bend points of the resistance curve are connected to fibre fractures. In this case the load appearing on the specimen drops dramatically. On the other hand a steep rise in resistance is connected with delamination and fibre pull-out. This result proves that a change of the load always leads to a change in electrical resistance.

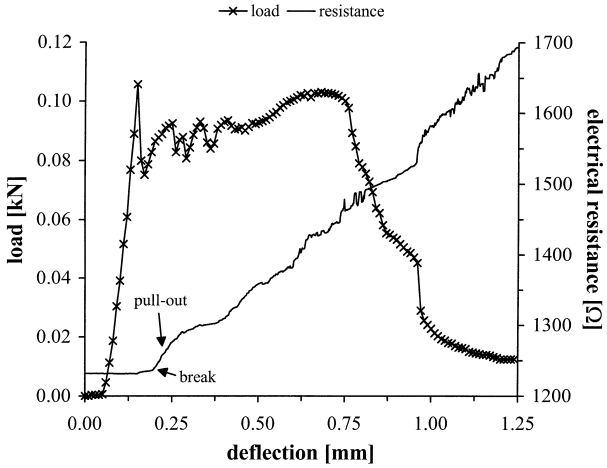

Fig. 6. Load-deflection curve and electrical resistance when applying bending load to a specimen made of Tyranno 1 fibres and Supremax® by slurry infiltration.

4 Conclusion

The relation between electrical properties, mechanical strength and heat treatment parameters for SiC-fibres was determined. Moreover in situ detection of the electrical resistance under applied mechanical load in electrically conductive composites was executed.

A correlation between the electrical resistivity and the mechanical strength of the fibres was found and an evaluation of the resistivity after the composites' production can be carried out with the dependencies found. The *in situ* detection of the electrical resistance was possible and the results show that the strain and the development of damage due to fibre fracture and delamination can be detected.

Acknowledgements

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