Transparent glass fiber/glass matrix composites

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Abstract: Nextel 440 fibers were coated with boron nitride (BN), titaniumoxide (TiO_2) and both boron nitride and titaniumoxide in succession by CVD. The fibers were characterized by single fiber tensile testing before and after heat treatment. Two different glass matrices, TELUX 756k and SCHOTT F4, were reinforced with the fibers. In the case of boron nitride single coatings and boron nitride-titaniumoxide double coatings the reinforcement leads to an improvement of fracture toughness, mechanical strength and the composites are slightly transparent. Nearly 50% transmission was found for the titaniumoxide coated fiber in optically matched glass but the absence of fiber debonding results in brittle fracture behaviour of the composite.

1. Introduction

The mechanical properties of compact glass are determined by the "thermal prehistory" of the glass and the surface quality but they can clearly be enhanced by reinforcement with fibers. Besides the mechanical strength the fracture toughness and the thermal shock resistance are improved. As a result of differing optical properties of matrix and fiber the glass matrix composites known so far are not transparent to visible light. The aim of our work is to develop transparent reinforced glass. In order to achieve optically transparent reinforced glass the mechanical and optical properties of the glass and fiber have to be adapted. The following facts should be taken into account for the selection of these components: Thermal expansion matrix = thermal expansion fiber, Refractive index matrix = refractive index fiber, Softening temperature fiber > softening temperature matrix, YOUNG's modulus fiber > YOUNG's modulus matrix.

The improvement of mechanical strength and toughness correlates with different "reinforcement mechanisms".[1] Overloading of the material results in the matrix cracking and the load transferring onto the fibers. Total fracture is prevented by crack deflection along the fiber-matrix interface. Increasing stress leads to the debonding of the fiber within the matrix and the fiber is pulled out the matrix. So the fiber-matrix interface plays an important role in realising reinforcement mechanisms like crack deflection, debonding and pull-out.

2. Experimental

2.1. Used Composite Components

For the investigations presented here the following components were used (Table 1):

Table 1: Properties of the used composite components [2]/[3]

		Glass matrix		Fiber
		F4	756k	Nextel 440
Manufactured by		SCHOTT	TELUX	3M
Refractive Index n_D		1,61	1,49	1,616
Thermal expansion coefficient (20..300°C)	$[10^{-6}/K]$	9,1	4,8	5,3
Transformation temperature	[°C]	439	470	-
YOUNG's-modulus	[GPa]	56	45	190
Bending strength	[MPa]	48	52	-
Tensile strength	[GPa]	-	-	2,07

Nextel 440 is a nanocrystalline oxidic fiber consisting of 70% alumina, 28 % silica, and 2% boron oxide.

2.2. Interface design and fiber characterisation

Interfaces should act as a diffusion- and reaction barrier and prevent intermingling of fiber and matrix. From a mechanical point of view carbon layers with graphite similar plain structure have proven their usefulness.[4] From an optical point of view the graphite isomorphical hexagonal boron nitride seems to be suitable. Its transparency to visible light makes it useful as an interface in transparent fiber reinforced glass. Also oxidic interfaces, e.g. TiO_2, ZrO_2 or SnO_2, have been investigated, but in these cases the absence of a slide-plain crystal structure leads to the problem of the optimization of the interface morphology, i.e. the interfaces have to be nanocrystalline or amorpheous with a small surface roughness.

A chemical vapour deposition process is used to coat the fibers with the aforementioned substances.[5]/[6] The typical coating thickness of the interfaces lies in the region of 30 to 150 nm. The characterization of the fibers with and without coating was carried out among other things by single fiber tensile strength testing before and after heat treatment in air. The heat treatment simulates the manufacturing conditions and, respectively, higher temperatures in practical application.

No significant change of the single fiber tensile strength was found for desized and BN-coated (50, 100 and 150 nm coating thickness) Nextel 440 fibers after heat treatment over 5 hours up to 500 °C. A heat treatment up to 750°C leads to a small decrease in tensile strength for desized and coated fibers with 50 and 100 nm coating thickness. Fibers with 150 nm BN coating thickness showed a decrease down to 705 MPa. Similar results were found for Nextel 440 fibers with 30 and 90 nm TiO_2 coating. The results for the desized fiber and the 35 nm BN + 30 nm TiO_2 double coating are shown in figure 1. Both a double CVD coating process and the heat treatment do not affect the tensile strength in a significant manner. The TiO_2 coating was specified as anatase by RAMAN and XRD investigations and undergoes no modification changes under the described conditions.

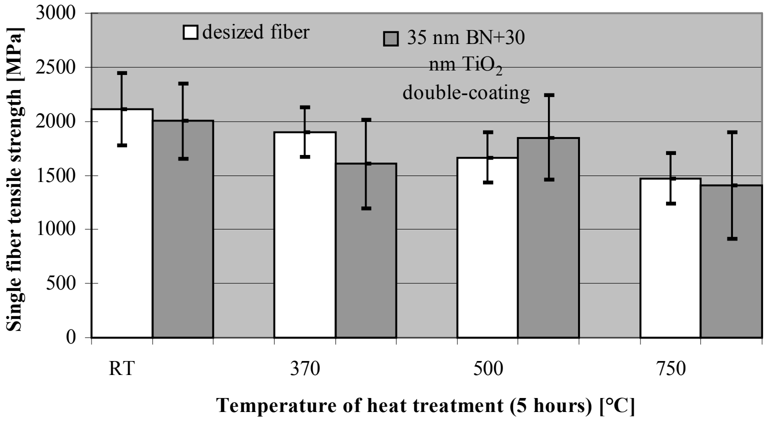

Figure 1: Single tensile fiber strength of Nextel 440 fibers

2.3. Manufacturing technology

The composites were manufactured by a common slurry infiltration process in aqueous solution. The received tapes were cut and stacked to prepregs. After drying these were hotpressed in vacuum.[7]

3. Results and Discussion

At first a Nextel 440 fiber-matrix combination with matched thermal expansion coefficients was used to investigate the fiber matrix interface and the coating influence on the mechanical properties of the composite. Therefore a common unleaded borosilicate glass, 756k, was choosen because of its use in earlier investigations.[8] Next an optical lead-silicate glass, F4, with adapted refractive index was combined with Nextel 440 fibers to study the influence of the interfaces on the optical properties. Unfortunately in this case the differing thermal expansion coefficients lead to strong thermal stresses. To avoid fiber-fiber interactions while studying interface influences the most composites were manufactured with a fiber volume content of only about 15% . For effective composites a fiber volume content of 30-40% is aimed.

3.1. Mechanical Testing

The results of mechanical testing (3-point bending test) are listed in table 2. All composites contain Nextel 440 long fibers in a 756k matrix and have an unidirectional fiber orientation.

Table 2: Mechanical characteristics of the composites in 756k matrix

coating/thickness	Desized	BN/40	BN/40	BN/94	BN/150	BN/150	BN/35+TiO_2/30
Volume content fibers [%]	15	15	30	15	15	30	6
Bending strength [MPa]	61 (±8,5)	92 (±0)	220 (±21,9)	110	98 (±4,5)	227 (±61,5)	99 (±17,7)
Fracture toughness [MPa*$m^{1/2}$]	1 (±0)	3 (±0,1)	6 (±0,1)	4	2 (±0,3)	6 (±1,3)	3 (±0)
Work of fracture [N/m]	71 (±21,5)	272 (±72,1)	866 (±53,0)	326	262 (±26,9)	854 (±343)	124 (±104)

The coating thickness of the BN layer has not shown any essential influence on the mechanical properties of the composites at the same volume content of fibers. The double coating achieved comparable mechanical values with lower volume content of fibers. SEM analysis of the fracture surface have proven effective reinforcement mechanisms (debonding and pull-out) both for single and double coatings (figure 2).

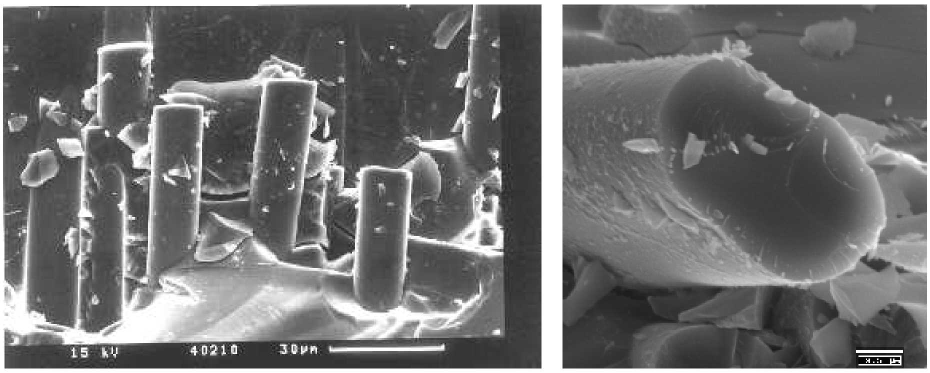

Figure 2: Fracture surface of a Nextel 440-756k composite with BN-TiO_2-interface (left) and a pull-out fiber in a Nextel 440-756k composite with 94 nm BN interface

3.2. Optical Properties

For comparision of the optical properties the transmission spectra of the hotpressed F4 glass and the composites with desized and TiO_2 coated (30 nm) Nextel 440 fibers were measured (figure 3). The BN coated fibers have shown chemical incompatibility under hotpressing conditions and therefore worse optical properties.

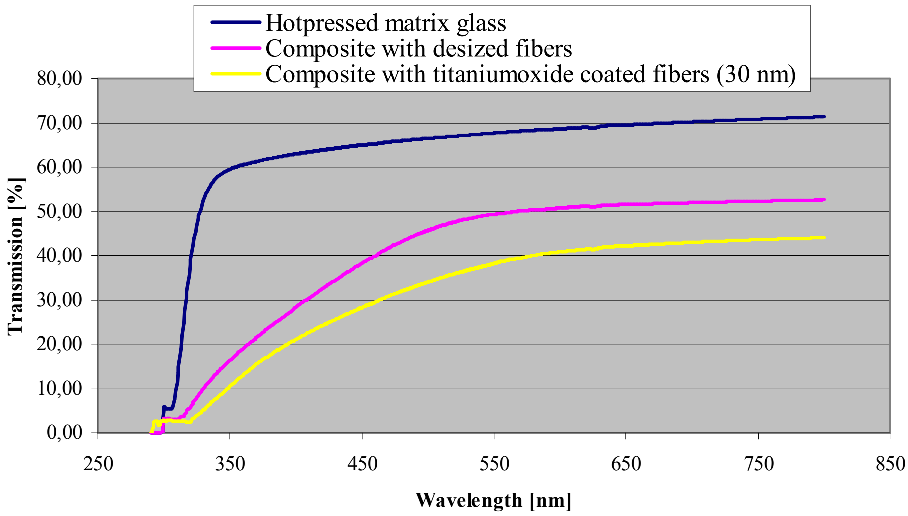

Figure 3: Transmission curves of F4 glass and composites with F4 glass, fiber content 15 vol-%, thickness: 1mm

In comparision with the pure hotpressed glass the fiber matrix interfaces reduce the transmission by about 30%. The 30 nm TiO_2 coating seems to have no evident influence compared with the desized fiber. The typical UV absorption at 385 nm for anatase , which is shifted to shorter wavelength by particle size reduction, was not detected owing to the low concentration.[9]

In the case of refractive index mismatch the fibers are clearly visible in the composite while the fiber matrix interfaces more disappeared when optically adapted components are used.

4. Conclusions

It was shown that it is possible to make transparent fiber reinforced glass by adapting the mechanical and optical properties of the components. BN single coatings and BN-TiO_2 double coatings, deposited on the fiber via a chemical vapour deposition process are good interfaces for realizing mechanically effective reinforcement mechanisms. The single fiber tensile strength of desized and coated fibers is not influenced significantly by heat treatment up to 500 °C.

Acknowledgement

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