HIGHLY POROUS SILICON CARBIDE PRODUCED BY SILICONIZING CARBON PREFORMS

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INTRODUCTION

Low density forms of carbon and ceramics find applications as thermal insulation materials, because of their low thermal conductivity and high thermal shock resistance. They also have other potential uses, e.g., as hot gas and molten metal filters, porous electrodes and catalyst supports. Because carbon can be manufactured in a wide variety of forms, it is an attractive precursor for forming ceramics, e.g., by reaction with Si, SiO, or SiO₂. In earlier papers we reported studies of the relationship between the structure and mechanical properties of open cell carbon foams [1] and SiC foams produced by direct siliciding of the carbon foams [2]. This paper reports a related study of the formation of low density SiC from a carbon bonded carbon fibre composite.

EXPERIMENTAL

The low density carbon was a commercial material manufacture by the Oji Paper Co Ltd., Japan [3]. A blended mixture of different types of chopped carbon fibres was dispersed in an aqueous slurry from which fibrous sheets were prepared using paper making technology. The fibrous sheets were impregnated with a resol phenolic resin and then laminated, hot pressed, and stabilised by heating in air. The laminate was then carbonised by heating to 1000°C at 125°C/h in nitrogen, followed by graphitization by heating to 3000°C at 500°C/h. The graphitised carbon preforms were heated at 2000°C in an atmosphere of argon containing Si vapour generated from Si₃N₄ [2]. Unreacted carbon remaining after the siliciding reaction was removed by oxidation in air at 800°C. The reaction products were characterised by optical and electron microscopy and X-ray diffraction. The carbon material, Figure 1, consists of laminated sheets in the XY plane

which have a near-random, planar array of chopped fibres with a small degree of preferred orientation in the paper run direction, X. Compressive strengths of the carbon and SiC materials were measured in the Z direction and flexural strengths were measured by bending rectangular specimens in the XY plane with the long axis in the paper run direction, X. Mechanical properties of the porous carbons were measured at room temperature; those of the SiC material were measured at room temperature and at 800°C in air.

RESULTS AND DISCUSSION

The unreacted carbon preforms have bulk densities of ~0.51 g cm⁻³, equivalent to a porosity of ~73%, Table 1. XRD spectrum of the material after the siliciding reaction and removal of unreacted carbon shows the presence of SiC and some free Si. Thermodynamic analysis [2] shows that under the reaction conditions condensation of liquid Si is expected, as the Si vapour pressure is supersaturated with respect to the liquid. SEM micrographs (not included in the Abstract) show that the fibrous nature of the carbon preform is retained, but the fibres have been converted to microcrystalline SiC, with a grain size in the range 2-20 microns, and some free Si. The volume expansion associated with the formation of SiC from C has been accommodated in the porosity of the preform. Complete conversion of the C to SiC would result in a reduction in porosity from 73% to 46%. The porosity in the SiC material is slightly higher than this value, Table 1.

In flexural tests both the carbon material and the SiC material show brittle failure modes. In compression, the SiC material shows a brittle crushing failure mode, Figure 2; the carbon material also shows a crushing failure mode in compression, but there is evidence for a shear component in the failure, probably as a result of its graphitic character.

The flexural strength of the material is increased by a factor of 2-3 on conversion to SiC and there is a considerable increase in flexural stiffness, Table 1. The values of the compressive properties of the porous carbon material are lower than the flexural properties, because the laminae are bonded together by resin carbon that is weaker than the fibres in the XY plane. Compressive properties are substantially improved on conversion to SiC. The mechanical properties of the porous SiC are retained up to at least 800°C in air, i.e., conditions under which the carbon preform is completely oxidised.

CONCLUSIONS

A low density carbon bonded carbon fibre composite was substantially converted to microcrystalline SiC (with some free Si) by direct siliciding, while retaining the highly porous and fibrous morphology of the precursor. The compressive and flexural mechanical properties of the material were substantially improved after conversion to SiC.

<u>Acknowledgment</u> We thank the Oji Paper Co. Ltd., Japan, for financial support and for provision of samples of the porous carbon.

REFERENCES

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TABLE 1.	Physical and	mechanical pr	operties of the	porous carbon	preform and SiC

Material	Carbon	SiC	SiC .
Maximum HTT	3000℃	2000°C	2000°C
Test temperature/°C	25℃	25℃	800°C
Bulk density/($g \text{ cm}^{-3}$)	0.512 ± 0.053	1.504 ± 0.046	-
Porosity/%	73.3 ± 0.3	49.4 ± 1.7	-
Flexural strength/MPa	13.7 ± 0.6	35.9 ± 1.3	36.6 ± 3.2
Flexural modulus/GPa	2.9 ±0.4	47.2 ± 1.3	47.2 ± 1.9
Compressive strength/MPa	0.88 ± 0.03	18.5 ± 2.8	19.7 ± 1.7
Compressive modulus/GPa	0.035 ± 0.002	0.47 ± 0.03	0.49 ± 0.03





Fig. 1 Schematic microstructure of porous carbon.

Fig.2. Compressive failure for porous SiC