

List of figures

	Page
Fig. 2.1: The relationship between the fracture toughness of the diamond-SiC composite and the grain size of its matrix	7
Fig 2.2: the process of infiltrating a preform with open porosity	9
Fig 2.3: Juncture of interfacial tensions for a liquid on a solid surface and cases of non-wetting (a) and wetting (b)	10
Fig 2.4: An illustration of Darcy's plot of infiltration height vs pressure	13
Fig 2.5: Self-diffusion coefficients of ^{14}C lattice diffusion (D_{1c}), ^{14}C grain-boundary diffusion (D_{bc}) and ^{30}Si lattice diffusion (D_{si}), as a function of $1/T$	19
Fig 2.6: Plot of the degree of reaction of diamond and silicon, α , vs time. Solid line represents the best fit of the Avrami-Erofeev equation to exponential data for the (111) reflection. The insert shows error bars in reaction rate, k , determination for the different reflections of SiC	21
Fig 2.6: The formation and lateral advancement of the SiC layer controlling the spreading of molten silicon as it happens	24
Fig 3.1: SEM micrographs of grade 1 diamond powder a) uncoated, b) coated with 10% resin after curing but before pyrolysis and c) coated with 10% resin after pyrolysis	32
Fig 3.2: SEM micrographs of grade 9 diamond powder a) uncoated, b) coated with 10% resin after curing but before pyrolysis and c) coated with 10% resin after pyrolysis	33
Fig 3.3: SEM micrographs of grade 16 diamond powder a) uncoated, b) coated with 10% resin after pyrolysis	33
Fig 3.4: SEM micrograph typical of the aluminum-silicon alloy as received from the manufacturer	34
Fig 3.5: SEM micrographs in secondary electron mode of a) the CVD diamond used for the investigation of the diamond/silicon reaction and for the determination of the wetting angles of the diamond/silicon system, b) the silicon wafers used for the investigation of the diamond/Si reaction	35
Fig 3.6: The temperature-time cycle used in pyrolysing the phenolic resin	38
Fig. 3.7: The average ratio of G-band intensity to diamond Raman peak intensity for the D2 and D9 diamond with initial 5 and 20% resin after their pyrolysis	39
Fig 3.8: The assembly of the preform and the silicon just before infiltration	40
Fig 3.9: Temperature/pressure cycle for infiltration of carbon-coated diamond with silicon.	41
Fig 3.10: Temperature/pressure cycle for infiltration with Al/Si alloy.	41
Fig 3.11: The set-up for the study of the reaction between diamond and silicon	42

Fig 3.12: An illustration of how the wetting angle was determined using a heating microscope	43
Fig. 3.13: The polishing of the samples using a resin-bound diamond wheel. The sample counter rotates against the wheel and a force is applied on the sample	45
Fig. 3.14: Wear test using a rotating cylinder of resin-bound epoxy flour conducted on the composite material produced	46
Fig 4.1: The wetting angle between silicon and a) diamond, b) graphite, as a function of temperature	48
Fig 4.2: The images showing the contact angle between diamond and silicon corresponding to a) 1000 °C, b) 1350 °C and c) 1502 °C	49
Fig 4.3: Images showing the contact angle between graphite and silicon, corresponding to temperatures at: a) 1000 °C, and b) 1500 °C	49
Fig 4.4: The wetting angle between carbon-coated diamond and silicon as a function of temperature	50
Fig 4.5: Images showing the contact angle between carbon-coated diamond and silicon, corresponding to temperatures at: a) 300 °C, b) 1150 °C, c) 1300 °C and d) 1477 °C	51
Fig 4.6: Backscattered SEM micrographs of the polished cross sections of the Diamond/SiC/ Si Interface as a function of reaction temperature and time	54
Fig 4.7: The influence of reaction time and temperature on a) the thickness of the SiC layer formed at the reaction interphase and b) the roughness of the SiC interface	55
Fig 4.8: Backscatter SEM micrographs of cross sections of the polished interface showing an eating into the diamond by the reaction at the SiC grain boundaries.	55
Fig 4.9: Backscatter mode SEM micrographs of etched cross sections of the sample reacted at 1475°C for 100 minutes at different places in different magnifications	56
Fig 4.10: SEM micrographs in SE mode of etched cross sections of the sample reacted at 1500 °C for 60 minutes (a,b,c), and 1450 °C for 15 minutes (d) showing the position of the groves and the cracks accompanying some groves (a)	57
Fig 4.10: A schematic showing the formation and growth of SiC a) in the starting period of layer formation and b) after the layer is formed	58
Fig. 4.11: The thickness of SiC layer formed from the reaction of Si with a) diamond (this study) and b) carbon (Zhou and Singh study) [4], as a function of reaction time	60
Fig 4.12: Self-diffusion coefficients of ¹⁴ C lattice and grain-boundary diffusion and ³⁰ Si lattice diffusion in β-SiC as a function of 1/T	61
Fig 5.1: A DTA pattern obtained on the heating, up to 1450 °C (heating rate of 2 °C/min) of an admix of diamond and Si powder	66
Fig 5.2: A typical micrograph in backscatter mode, of a sample after hot-pressing an admix of diamond and silicon from a micronizing mill at 1500 °C	68

Fig 5.3: A typical micrograph showing a porous material with WC contamination, of a sample after hot-pressing an admix from a planetary ball mill at 1550 °C (sample 3, Table 5.1)	68
Fig 5.4: A typical micrograph of a sample hot-pressed at 1550 °C	69
Fig 5.5: a) A micrograph typical of a fracture surface of a sample hot-pressed at 1500 °C (sample 8, Table 5.1), b) A close-up on same sample at 5000X	69
Fig 5.6: a) A micrograph typical of a fracture surface of a sample hot-pressed at 1500 °C (sample 9, Table 5.1) and b) a close-up of the boxed section in a).	70
Fig 5.7: XRD patterns for samples reacted at 1450 °C for 30 min followed by a heat treatment at 1550 °C for 60 min, a) heating rate of 5 °C/min (sample 3, table 5.1) and b) heating rate of 10 °C/min (sample 4, table 5.1).	71
Fig 5.8: An example of a microstructure of a product obtained through pressing with an SPS machine showing wetting by silicon but non-densification	72
Fig 5.9: An example of a microstructure of a product obtained through pressing with an SPS machine showing lack of silicon in areas of porosity (non-densification)	72
Fig 6.1: The eutectic-forming Al-Si alloy phase diagram	79
Fig 6.2: The Si-C-Al phase diagrams from fact sage program, a) at 1000°C and b) at 1500°C, showing the dependence of SiC formation in the presence of Al	80
Fig 6.3: The free enthalpies of formation of SiC and Al ₄ C ₃ against temperature calculated using fact sage program, normalized on cations	81
Fig 6.4: A DTA pattern obtained on the heating, up to 1500 °C of an admix of diamond and the Al/Si alloy powders	82
Fig 6.5: An SEM micrograph of the loose diamond infiltrated with the Al/Si alloy (sample 1 in table 6.1) using a pressure of 20 MPa at 1000 °C for 1 hour	85
Fig 6.6: SEM micrographs of the sample infiltrated at 1000 °C (sample 2, Table 6.1) at a) the centre of the infiltrated area, b) the top of the infiltration and c) the bottom	86 87
Fig 6.7: SEM micrographs of the interfaces between the alloy and the diamond of samples infiltrated at a) 1000°C (sample 2, Table 6.1) and b) 1500°C (sample 4, Table 6.1). c) shows the details of interface in a).	
Fig 6.8: A SEM micrograph typical of a compacted pyrolysed diamond preform showing one of the large pores to be about 55 μm	88
Fig 6.9: SEM micrographs of a resin-bound diamond preform (sample 5 in table 6.1) at different magnifications	88
Fig 6.10: A typical fracture surface micrograph of sample 9 showing phases after reaction-infiltration of diamond with Al/Si alloy at 1500°C for 30 minutes	89
Fig 6.11: Typical XRD patterns of the materials infiltrated at 1000°C a) non-preformed diamond with diamond peak labelled C (sample 2) and b) preformed diamond (sample 5)	90
Fig 6.12: X-Ray diffractogram of sample 2 (Table 6.1) after heat treatment at 1300 °C for one hour	91

Fig 6.13: X-Ray diffractogram of sample 8 (Table 6.1) infiltrated at 1000°C (1 hr), followed by reaction at 1300°C (30 min).	92
Fig. 7.1: The pore size distribution of preforms a) Grade 1 (D02) with varying resin content, b) Grade 9 (D09) with varying resin content, and c) 10 wt% resin in grade 1, 8 and 22	98
Fig. 7.2: Some cross-sections showing the maximum infiltration heights after infiltration at 1500 °C for 30 minutes of grade 1 diamond with a) 5 %, b) 10 %, and c) 20 % resin	101
Fig. 7.3: A cross-section of grade 9 diamond with 5% resin showing the maximum infiltration height after infiltration at 1500 °C for 30 minutes	102
Fig. 7.4: Typical backscatter SEM micrographs of polished cross-sections of grade 16 diamond (5% resin) preforms infiltrated for 30 minutes at a) 1450 °C and b) 1500 °C	103
Fig. 7.5: Typical backscatter SEM micrographs of polished cross-sections of grade 9 diamond (10% resin) preforms infiltrated for 30 minutes at a) 1450°C and b) 1500°C	104
Fig. 7.6: The spot EDS results of a polished sample produced at 1450 oC (fig 7.4a) showing the white phases to be contamination by metals, Fe, Zn, Ni, Cr and oxygen	104
Fig. 7.7: Typical backscatter SEM micrographs of a polished cross-section of grade 1 diamond (10% resin) preform infiltrated for 30 minutes at 1500°C	105
Fig. 7.8: A comparison of the wear behaviour of the composite material made against that of Element Six Syndax-material	106
Fig. 7.9: A comparison of SEM micrographs of a) Syndax and b) the material	107
Fig. 7.10: SEM micrographs of the wear scars of a) the composite material made at different magnifications and c) syndax at different magnifications.	108
Fig. 7.11: Typical optical images obtained after indentation for hardness measurements	109
Fig. 7.12: Infiltration rate constant (m ² /s) vs pore diameter as defined by 20%, 50% and 80% total infiltration volume	111
Fig. 7.13: Infiltration at 1500 °C for 30minutes a) A plot of infiltration heights, b) A plot of the calculated melt flow rates (see appendix) against pore size for the different diamond grades	113
Fig 7.14: Time dependence of silicon conversion from carbon/Si reaction	116