

Si-SiC ceramics from plant precursor

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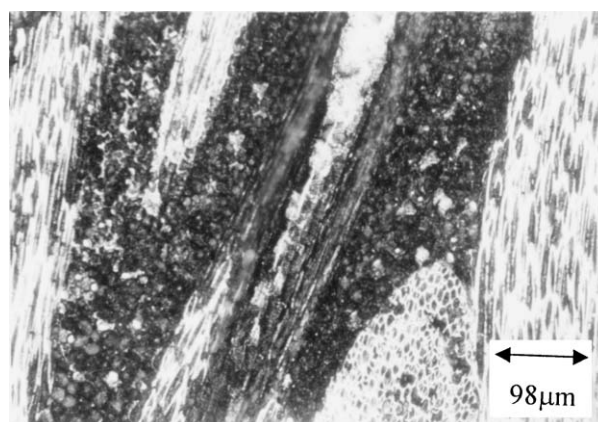
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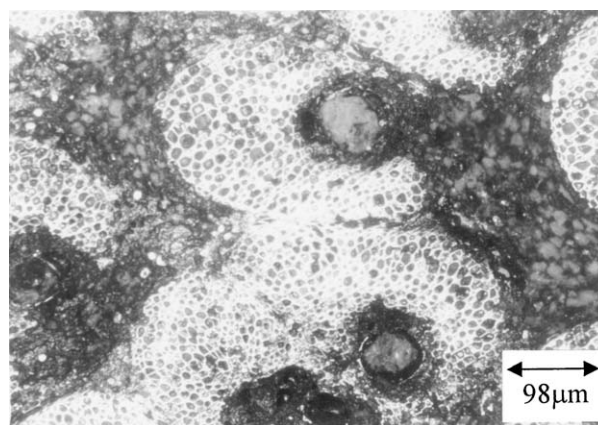
Synthesis of materials from naturally grown plants has recently received interest [1, 2]. Plants often possess natural composite structures and exhibit high mechanical strength, low density, high stiffness, elasticity and damage tolerance. These advantages are because of their genetically built anatomy, developed and matured during different hierarchical stages of a long-term evolutionary process. There is a possibility of producing novel silicon carbide (SiC) ceramic materials nearly isomorphous to naturally grown plants, on the macro- and micro scale. SiC ceramics are commonly utilized for different structural applications. Among the various methods of producing SiC ceramics, reaction bonding/reaction sintering [3] has been shown to be the cheapest and the most commercially viable. Reaction bonding/reaction sintering is based on the C-Si reaction and is conventionally restricted to synthetic preforms [3, 4]. SiC ceramics can also be made using bio-structure derived preforms [5–7]. A wide variety of plants and plant-parts can be used for making different varieties of SiC ceramics [8–10]. Biological preforms from various soft woods, hard woods and non-wood ingredients, commonly used in pulp and paper manufacturing, can be employed for producing SiC ceramics. In view of the variations in dimensions, composition and morphology of the naturally grown plant structures, the shape and composition of the bulk SiC produced will vary significantly. The formation of SiC ceramics using monocotyledonous trees as the precursor is reported here.

A monocotyledonous tree from a local source was used as the precursor plant. It was transformed to a porous skeletal carbonaceous preform of rectangular shape (of approximately 41 cm² external surface) following a pyrolysis process at around 800 °C without any structural damage (cracking, loss of integrity etc.) [11]; it was subsequently reacted with silicon to yield Si/SiC ceramics under vacuum at a temperature of around 1600 °C [12]. After the reaction the macroscopical structural integrity was totally retained. The carbon preform and the final product were subjected to: X-ray diffraction (PW 1710, Philips, Holland); microstructural examination using light microscopy (Zetopan, Reichert, Austria) and scanning electron microscopy (SE-440, Leo-cambridge, Cambridge, U.K.); determination of the volumetric phase composition of the final ceramic by the point counting method from light photomicrographs; and thermogravimetric

analysis (TGA) using a thermo-balance (STA 490C, Netzsch-Geratebau GmbH, Germany) up to 1200 °C in flowing air (70 ml/min) at a rate of 10 °C/min. Density was determined by water displacement method and porosity by the boiling water method. The three-point bending strength and Young's modulus of Si/SiC ceramic material were determined at room temperature using an Instron Universal Testing Machine, and the deflection was monitored through a LVDT with a resolution of 0.05% of the full scale deflection. The specimens were 45 mm × 3.5 mm × 2.5 mm in size, ground and polished to 1 μm finish. Five tests were conducted and an average value was recorded.



(a)



(b)

Figure 1 Light photomicrograph of the carbon preform: (a) longitudinal view and (b) cross-sectional view.

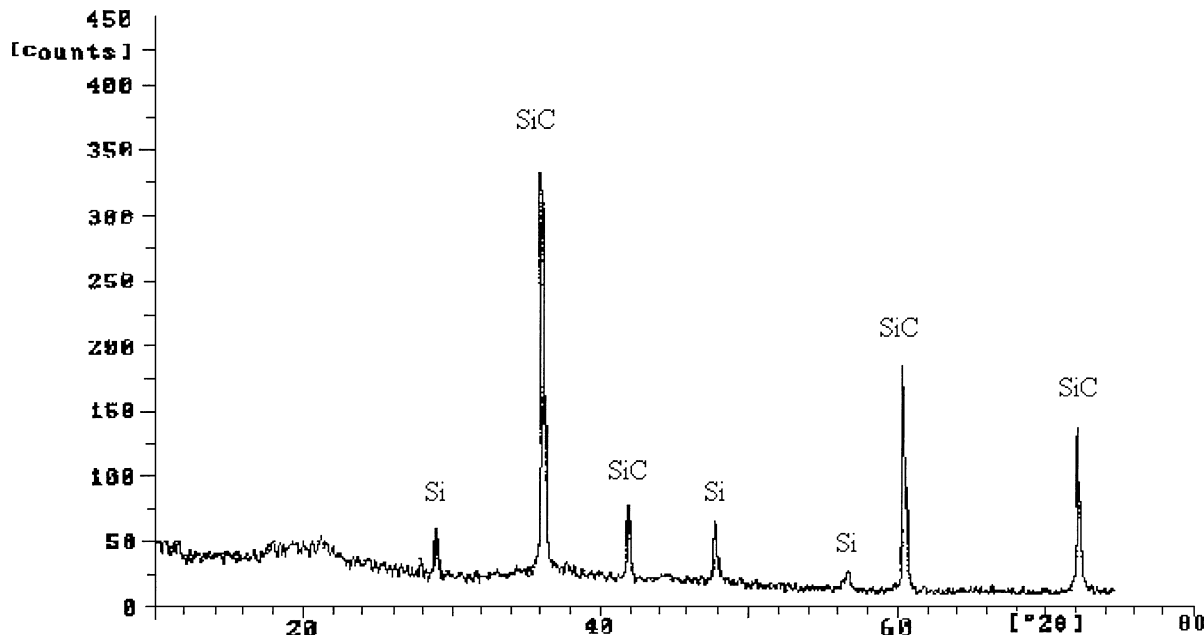


Figure 2 XRD profile of Si-infiltrated carbonaceous preform from plant, showing the presence of Si and β -SiC.

The transformation to the carbon preform was found to be associated with an overall change in linear dimension of around 29%. Despite such a vast change, the porous macro-structure of the precursor plant was retained with high precision. The microstructure of the carbon preform shows the retention of tubular elongated cell structures of the precursor plant, which are aligned with the axis of the tree (Fig. 1a). Hollow channels of varying diameters, originating from the tracheal pores, are seen to be distributed in the cross-section (Fig. 1b). The transformed preform contained 35.7 vol% solid, which is essentially amorphous carbon. A marginal change in the overall linear dimension of the Si/SiC material, derived from the carbon preform, was noticed. The density and porosity of the final ceramic were found to be 2.7 gm/cm^3 and 0.7 vol% respectively. The XRD profile (Fig. 2) of the material shows the presence of β -SiC and Si.

After the reaction the carbonaceous pore wall is converted to β -SiC and the pores are filled with unreacted Si; the composition of the final ceramic was found to be 43.6 vol% SiC and 55.7 vol% Si. The thickness of the β -SiC layer and the diameter of the Si-filled pores were found to vary. The retention of the tubular elongated cell structures is clearly seen in the converted specimen (Fig. 3), the thickness of β -SiC layer and the diameter of Si-filled pores being $9\text{--}13 \mu\text{m}$ and $7\text{--}8 \mu\text{m}$, respectively. Converted β -SiC structures and the Si-filled channel pores are fibrous in shape. When viewed under SEM, the preservation of the cellular ring morphology is distinctly evident (Fig. 4); formation of around $5 \mu\text{m}$ thick SiC cellular ring around a Si-filled pore of around $53 \mu\text{m}$ diameter is visible.

The dense Si/SiC composites are found to have average flexural strength and Young's modulus of 264 MPa and 247 GPa respectively, at room temperature. The strength and elastic modulus data are comparable to those of the conventional RBSiC ceramics [13]. The presence of fibrous structures is presumed to be contributing to the strength of the final material. Thermo-

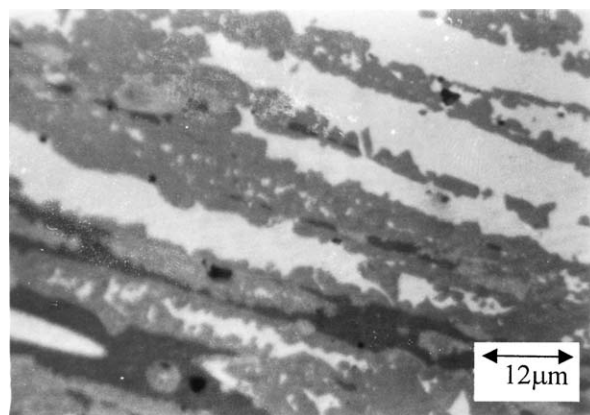


Figure 3 Light photomicrograph of Si/SiC showing near-isomorphism with precursor plant.

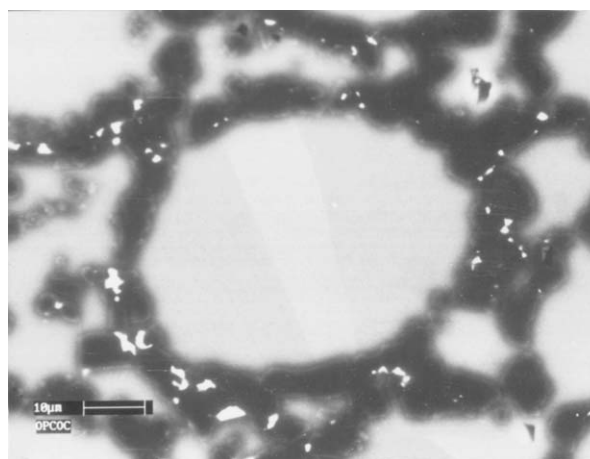


Figure 4 SEM photomicrograph of Si/SiC showing formation of β -SiC cellular ring structure around Si-filled pore.

gravimetry of the composite ceramic (Fig. 5) exhibits slow and small decrease in weight ($\sim 9\%$), indicating sufficient resistance of the final ceramic in a high temperature oxidative environment.

The present study demonstrates the possibility of producing novel Si/SiC ceramics using a

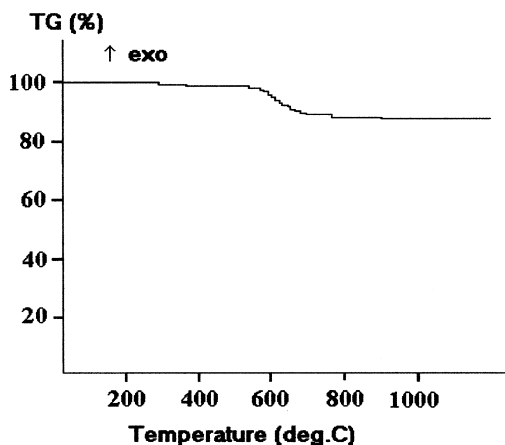


Figure 5 TGA scans during heating to 1200 °C of Si/SiC showing slow and low weight loss.

monocotyledonous tree of local and renewable source as the readily machinable precursor. The final material has little porosity and macro- and microstructures nearly isomorphous to the parent plant. The end ceramic has room temperature flexural strength and Young's modulus of 264 MPa and 247 GPa respectively, comparable to those of the conventional RBSiC. Presence of fibrous structures may be contributive to the adequate strength of the material. The Si/SiC ceramic material shows high oxidation resistance during heating to 1200 °C in flowing air.

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