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Feature article

Effect of degree of crystallinity on elastic properties of silicon carbide fabricated using polymer pyrolysis

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ABSTRACT

In this study, silicon carbide (SiC) is fabricated using polymer infiltration and pyrolysis (PIP). Allylhydridopolycarbosilane (AHPCS) is used as the preceramic polymer. Final processing temperatures are varied to observe the change in microstructure as well as physical and mechanical properties. Density, porosity and thermal conductivity are determined as a function of processing temperatures. Microstructural characterization, done using atomic force microscopy (AFM) and X-ray diffraction (XRD), has revealed the presence of nanocrystalline domains with grain sizes in the range of 5–20 nm. The degree of crystallinity is determined using non-contact mode atomic force microscopy. The degree of crystallinity follows an increasing trend with processing temperature. Hardness and modulus are determined using nanoindentation, and are found to be influenced by the degree of crystallinity.

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1. Introduction

Due to its superior qualities and ease of processability, AHPCS has been extensively used as a matrix source for polymer precursor derived silicon carbide (SiC) composites. It has also been used for fabricating SiC coatings and for joining ceramic parts [1]. However, for determining the feasibility of using precursor derived SiC for various applications, extensive mechanical characterization is required, especially as a function of processing conditions. Although AHPCS has been used for number of applications, limited mechanical property data for AHPCS derived SiC has been available through open literature till date [1–4].

Polymer to ceramic conversion is accompanied by large amount of volume shrinkage that results in cracks and pores in bulk samples. Since cracks and pores highly influence bulk properties, to get true local property for AHPCS derived SiC, their nano-scale characterization is important. As mechanical properties such as elastic modulus, and hardness depend on microstructure, evaluation of changes in microstructure with processing conditions is also of great importance.

There have been very few studies on bulk mechanical characterization of AHPCS derived SiC [1–5]. One of the reasons could be that volume shrinkage during polymer to ceramic conversion makes it difficult to fabricate monolithic specimen for bulk characterization. However, mechanical properties, if available, would be of great value for designing ceramic parts and designing composites using AHPCS derived SiC as matrix. Interrante et al. fabricated SiC/SiC composites using AHPCS and determined the average fracture stress to be ~378 MPa which was 80 MPa higher than other reported values [2]. Moraes et al. reported the effect of heat treatment for a range of temperature on fracture toughness and Vickers hardness for AHPCS derived SiC using PIP technique [1]. A non-linear relationship was found for both fracture toughness and Vickers hardness with heat treatment. Recently, Zunjarrao et al. have reported elastic modulus and hardness of AHPCS derived SiC after one cycle of PIP processing [3]. The highest modulus and hardness values were observed for SiC processed at 1150 °C, and were found to be 218 and 30 GPa, respectively.

Since AHPCS derived SiC has been evaluated as a potential candidate for structural applications as well as matrix for nuclear fuel [6], physical properties like thermal conductivity and density are also of great concern for increase in thermal efficiency. Moraes et al. reported increase in density with increasing processing temperature for SiC derived from AHPCS [1]. Dong et al. studied mechanical behavior of SiC/SiC which has AHPCS derived matrix and found that higher strength of SiC/SiC composite could be obtained with higher density [4]. The average strength for the composite from a three point bend test was found to be 400 MPa. According to Kotani et al. oxidation resistance of AHPCS derived SiC/SiC composite was better as compared to polycarbosilane (PCS) derived SiC/SiC composites [5]. Lee et al. evaluated thermal conductivity of SiC particulate reinforced AHPCS derived SiC composites [7]. Higher crystallization

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temperature resulted in improved thermal conductivity by removing carbon impurities and increasing crystallite sizes.

For any given ceramic, properties are governed by underlying microstructure. For understanding the property dependence on microstructure, it is important to follow microstructural evolution of AHPCS derived SiC. Nonetheless, only limited microstructural characterization data for AHPCS derived SiC exists in literature. Interrante et al. have studied conversion of AHPCS to amorphous SiC in a temperature range of 800–1000 °C using a combination of differential scanning calorimetry (DSC), thermo gravimetric analysis (TGA), X-ray diffraction (XRD), transmission electron microscopy (TEM), Raman spectroscopy and solid state nuclear magnetic resonance (NMR) [8]. Very small amount of mass loss was detected using DSC/TGA analysis and oxygen in the form of SiO_xC_y in amorphous SiC was detected using elemental analysis. Microstructure of polymer derived SiC has been found to vary with different processing temperatures. For example, at lower processing temperature up to 1000 °C polymer-to-ceramic conversion results in amorphous SiC. Nano-crystalline domains of SiC start to form as the processing temperature is increased beyond 1100 °C [9]. Zunjarrao et al. have used XRD for determining the crystal size for AHPCS derived SiC processed at different temperatures [3]. Average crystallite size of 11 nm was observed for material processed at 1650 °C. Hardness and modulus were greatly influenced by microstructural change. However, the effect of nanocrystalline domain in amorphous matrix on the mechanical properties has not been probed from the degree of crystallinity perspective.

In this investigation, non-contact mode atomic force microscopy image analysis is used for determining the degree of crystallinity in polymer derived SiC as a function of processing conditions. In addition, the effect of nanocrystalline domains in an amorphous matrix on mechanical properties is investigated using a particulate composite model.

2. Experimental procedure

2.1. Bulk sample fabrication

Allylhydridopolycarbosilane has a nominal structure of $[Si(CH_2CH{=}CH_2)_2CH_2]_{0.1}[SiH_2CH_2]_{0.9}$ [1]. The polymer is designated as SMP-10 by the manufacturer (Starfire Systems Co., Malta, NY, USA) and is in the form of a clear amber colored viscous liquid at room temperature. Fabrication of SiC as a bulk sample purely from AHPCS is tricky and lengthy since shrinkage occurs as the precursor converts to ceramic SiC. The shrinkage is due to removal of oligomers and gaseous by-products during polymer-to-ceramic conversion. Using reinforcing agents such as SiC whiskers or fibers helps in minimizing the overall volume shrinkage. These fillers can be added in a considerable volume fraction in the composites and do not undergo a volumetric change during processing. However, the current investigation is focused only on the characterization of SiC derived from AHPCS. So, reinforcing agents such SiC fibers or whiskers were not used. Instead, SiC powder produced by AHPCS was itself used as filler.

Fig. 1 shows the flow diagram of complete PIP processing steps. This process requires heating of the polymer precursor to a temperature above 800 °C under controlled atmosphere for polymer to ceramic conversion. An inert atmosphere was maintained using a constant flow of ultra high purity argon (99.999%) to prevent oxidation of SiC at higher temperatures. The fabrication process was started by heating the liquid polymer precursor (AHPCS) to 650 °C with a heating rate of 1 °C/min and holding at that temperature for 10 min. This started the cross-linking of the polymer precursor. For complete conversion to SiC ceramic, the heating was continued till the desired final temperatures, i.e. 900, 1150, and 1400 °C

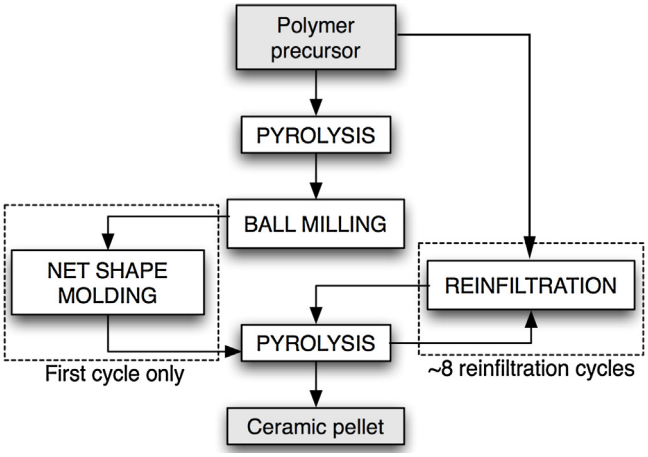

Fig. 1. Flow diagram of bulk SiC sample fabrication using polymer infiltration and pyrolysis.

with a heating rate of 3 °C/min. All specimens were held at the final temperature for 1 h to ensure thermal equilibrium and complete processing. Finally, the specimens were cooled down to room temperature at a cooling rate of 5 °C/min.

Due to the release of hydrogen gas and oligomers during the process, the acquired materials contained large voids. To produce a bulk sample with minimal porosity, these were ground using a hand grinder till the particles passed through a colander of mesh size 12. Subsequently, the particles were ground into a fine powder using a planetary ball mill (PM-100, Retsch GmbH, Haan, Germany) in a tungsten carbide (WC) bowl with 10 mm WC balls for 12 min at 300 rpm. These powders were used as reinforcing fillers in the next step of the fabrication process.

The powders were mixed with a small amount of polymer precursor (3% by weight of the milled powder) to prepare a semi-dry paste. A steel ram–cylinder setup was used to compact the semi-dry paste to a cylindrical preform. Hand compaction was used to apply a maximum load of ~450 N. The resulting plugs were then heated to the final temperature (900, 1150, or 1400 °C) using the heating cycle described earlier. Although the resulting samples had a good dispersion of SiC particles in an amorphous silicon carbide matrix; pores were present in the plugs due to the release of the hydrogen gas and oligomers during polymer pyrolysis. Multiple PIP cycles were then carried out to minimize porosity and to increase the material density. The infiltration with the liquid preceramic polymer was carried out under vacuum with a repeated 1 h cycle for 4 h and 1 min purge in between of each cycle.

The plugs were cut into 1 mm thick discs using a precision sectioning saw (Isomet 1000, Buehler, 202 Lake Bluff, IL, USA) after the 3rd PIP cycle. Since, 8 infiltration cycles are sufficient to obtain the maximum achievable density [10], the discs were subjected to infiltration and pyrolysis till the 8th cycle. Slicing the plugs into discs allows for more efficient reinfiltration process.

2.2. Physical and thermal characterization

Density and porosity of the silicon carbide samples were determined after the 8th cycle of infiltration and pyrolysis as a function of different processing temperatures. The buoyancy method was used for determining bulk density and open porosity of the samples using a density measurement kit along with a high-resolution analytical balance [11]. Then, the samples were crushed and a helium pycnometer (Ultrapycnometer 1000, Quantachrome Instruments, Pittsburgh, PA, USA) was used for measuring solid density. The closed porosity was calculated from the measurement of bulk and solid densities.

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