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Exfoliation-assisted dispersion of short carbon fibers in silicon carbide powder

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ABSTRACT

This study brings out the findings of milling and dispersion of carbon fibers (CFs) and silicon carbide (SiC) powder in a wide range of CF to SiC ratios (10:90, 20:80, 30:70, and 50:50 vol%). The composite powders were prepared by chopping and exfoliation of CFs, and ball milling of CFs and SiC powder in isopropyl alcohol. The exfoliation of CF tows resulted in an excellent dispersion of CFs in SiC powder without any dispersing agent for all CF loadings. However, the distribution of CFs in SiC for the samples with 10 and 20 vol% of CFs was found extremely uniform compared to those with 30 and 50 vol% of CF loadings. Furthermore, the exfoliation of CF tows yielded excellent dispersion and distribution of CFs in SiC without damaging the surface of CFs. The results further showed that the average particle size (APS) of composite powders increased with CFs loading.

1. Introduction

In the last few years, carbon/silicon carbide composites (C/SiC composites) have attracted considerable interest for applications in a wide temperature range due to their excellent properties at elevated temperature such as strength, stiffness, oxidation resistance, low density, creep resistance, chemical stability, and low wear [1]. Because of their excellent set of properties, they are best suited for aerospace applications such as wing leading edges [2], strut leading edges, BL splitter, combustor segments, etc. Apart from aerospace applications, they have important applications in the field of high-temperature sensors, optoelectronics, and brake discs [3,4].

The C/SiC composites are a special class of advanced materials that are ceramic in nature. In this material, either carbon or silicon carbide acts as reinforcement. Basically, two systems exist for C/SiC composites: (1). Carbon as a reinforcement and silicon carbide as a matrix, and (2). SiC as reinforcement and carbon as a matrix [5]. In both systems, either short fibers or continuous fibers (in the form of 1D, 2D, and multi-D woven cloth) act as reinforcement. Since SiC fibers are brittle, their use as reinforcement is seldom done. The usage of CFs, carbon whiskers, particulates, etc., as reinforcement for C/SiC composites is very common. Over the past years, various kinds of C/SiC composite have been fabricated. Among them, SiC is heavily used as a matrix whereas particulates, whiskers, and fibers of carbon are employed as reinforcement. CF reinforced SiC matrix composites can be fabricated by the application of any one or a combination of two or more than two of the following processes:

1. Chemical vapor infiltration
2. Polymer infiltration and pyrolysis
3. Reaction sintering
4. Hot pressing

Although C/SiC composites have been extensively studied by various researchers, much focus is given for continuous CF reinforced SiC matrix composites [6–9]. The use of short CFs as reinforcement is a way to reduce the cost of the composites. The short fiber reinforced composites are increasingly used in a wide range of applications because of their easy adaptability to conventional manufacturing techniques and ease in fabrication [10]. In short CF reinforced SiC matrix composites system, the properties of the composites can also be tailored to a large extent as per requirement. This tailorability and the end properties of short CF reinforced C/SiC composites depends upon various parameters. The dispersion and distribution of CFs in SiC matrix and the APS of the charge (CFs and powdered SiC) are some of such parameters. The agglomeration free distribution of CFs in SiC matrix plays key role in interphase bonding, crack propagation, mode of failure, etc. The particle size of the starting materials has long been recognized as a key factor in the end properties of a material. The packing density increases with a decrease in particle size. Both agglomeration free distribution of CFs in SiC powder and control over APS are very complex due to differences in the nature and density of CFs and SiC.

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A good amount of work has been done for the fabrication of short CF reinforced SiC composites by varying processing conditions, sintering aids, and the ratio of reinforcement to matrix [10–14]. Furthermore, some work has been done on the dispersion of CFs in ceramic powders using dispersing agents [15–18]; even then, much focus on this area has not been given. Additionally, the usage of various kinds of dispersing agents by various researchers could not result in uniform dispersion of CFs in the order of 50 vol% of CFs in SiC [19]. In addition to contamination due to the application of dispersing agents, the dispersion time of these reported techniques is quite high. The increased amount of time they employed resulted in damage to CFs [19]. In a nutshell, the present scenario on the dispersion and distribution of CFs in SiC powder provides a good scope for the usage of innovative and advanced techniques to get damage and dispersing agent free dispersion and distribution of CFs in SiC powder in a short span of time.

Furthermore, to the best of the authors' knowledge, work has not been done on studying the agglomeration free distribution of CFs in SiC powder using the concept of exfoliation of CFs prior to ball milling. Hence, efforts were made to study the influence of exfoliation on the dispersion and distribution of CFs in SiC powder through wet milling of exfoliated CFs in SiC powder. This paper briefs the study carried out on exfoliation-assisted dispersion of CFs in SiC powder in a wide range of CF to SiC ratios (10:90, 20:80, 30:70, and 50:50).

2. Experimental plan

2.1. Materials

PAN based CFs (T-300, 3 K, Torayca Co., Japan; 3 mm length) and SiC powder (7.361 μm APS, α-grade) were taken for the milling experiments. Isopropyl alcohol (LR grade) was employed as a slurry media for milling SiC powder and CFs. The samples with 0, 10, 20, 30, and 50 vol% of CFs were designated as TSM-00, TSM-01, TSM-02, TSM-03, and TSM-05, respectively.

2.2. Fiber chopping

The continuous CFs from the fiber spool were chopped into discrete length CFs using in-house designed and developed fiber milling equipment (Fig. 1) and method of un-interrupted fiber milling. The discrete CFs of 3 mm length were obtained using fiber milling equipment.

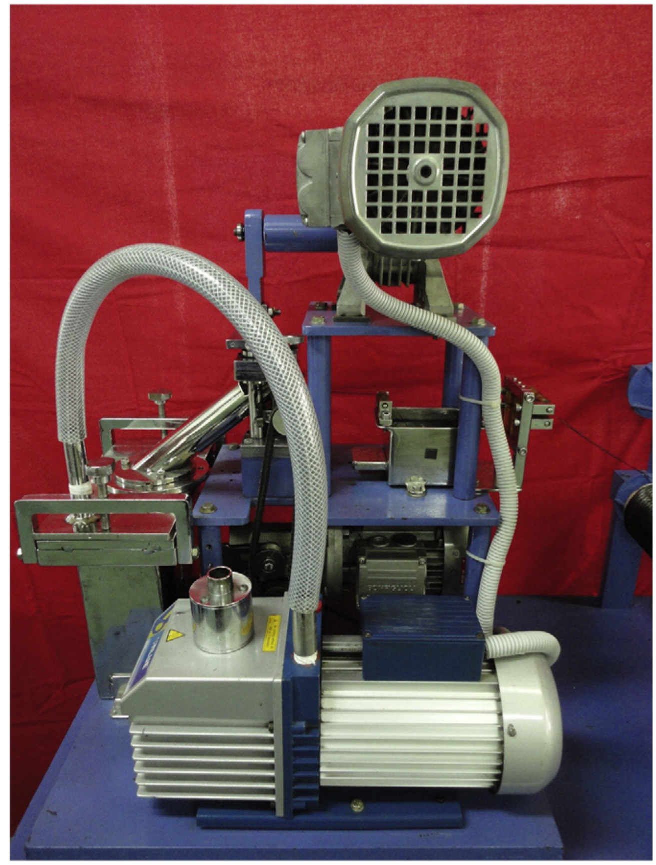

Fig. 1. Digital image of fiber milling equipment.

2.3. Grinding

The chopped CFs of 3 mm length obtained from fiber milling equipment were ground using a domestic mixer-grinder (Sumit make). Grinding is basically a size reduction operation. It employs attrition and impact mechanism for reducing the size of the solids/powders. Attrition means a reduction in size by rubbing with solid jar's wall, blades, etc. Impact means a reduction in size by impact or direct hit of solid blades on the surface, which is to be reduced. Grinding was done for a total of 4 min in 16 cycles of 15 s each.

2.4. Milling

The ball mill (Fritsch make, Pulversette P5 model) was employed for the milling study. The bowls (SS with inner coating of silicon-nitride) of size Ø 74.5 mm × 70 mm and the balls (Small ball of Ø 10 mm and big ball of Ø 20 mm) were used for this operation. The material of construction of balls was also silicon-nitride. Milling is basically a size reduction operation like grinding. Unlike grinding, it is used for getting a very fine particle size.

The ground exfoliated CF tows were dispersed in isopropyl alcohol (20 ml per 1 cm^3 of charge) with SiC powder. The powder mixtures were milled for 4 h in 8 cycles (30 min operation per cycle) with a soaking time of 5 min after each cycle. The main objective of giving residence time in between cycles is to minimize the heat generated during milling and to protect the material from any damage due to overheating.

2.5. Air drying

The electric air oven (Thermosystem make) capable of maximum heating up to 300 °C was used for the air-drying operation. The slurries of the samples were dried for 8 h at 90 °C at 5 °C/min. The drying operation was basically carried out to evaporate isopropyl alcohol from the slurry of CFs and SiC.

2.6. Crushing

After air-drying, the powder mixtures were found to have certain agglomerates. To break down these agglomerates to a finer scale, the powder mixtures were crushed using a domestic mixer-grinder in 4 cycles. In each cycle, crushing was carried out for 15 s in pulse form to avoid the wear on blades due to SiC powder.

2.7. Microstructure

After the crushing operation, the powders from all the samples were characterized for the morphology of CFs and SiC powder and for the distribution of CFs in SiC powder. The microstructure analysis of samples was carried out using Carl Zeiss make SMT EVO 50 model scanning electron microscope (SEM). SEM images were taken under variable pressures using 80P air pressure. A filament of LaB6 was employed for taking the images.