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Original Research Paper

Effect of milling parameters on exfoliation-assisted dispersion of short carbon fibers in silicon carbide powder

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ABSTRACT

A novel methodology for the dispersion of carbon fibers (CFs) in silicon carbide (SiC) powder without any dispersing agent was explored. Short CFs were chopped and exfoliated. The exfoliated fibers were dispersed in SiC powder through wet ball milling, drying and crushing. The influence of various ball milling parameters like milling time (MT), ball to powder ratio (BPR), slurry media to powder ratio (SMPR), etc., on the dispersion and distribution, and average particle size (APS) of composite powder was studied. Two MTs (4 and 8 h), two BPRs (4 and 8), and four SMPRs (6, 10, 20 and 35 mL/cm^3) were used. The dispersion and distribution was checked through environmental scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM). The results showed that the increase in MT and BPR improved the distribution along with dispersion but, surprisingly, infuse fiber filaments. The APS of powder mixture was found to decrease with MT and BPR. The results further showed that the dispersion and distribution was improved with decrease in SMPR. The severe ball formation was noticed at a SMPR of 35 mL/cm^3. Further, the APS was found highly dependent on SMPR.

1. Introduction

Ceramics are good choice for high temperature applications because of their excellent high temperature stability in oxygen atmosphere. In addition to this, their mechanical, thermal and chemical stability are very much promising. Various types of ceramics have been studied and still being studied for this type of applications. Although their metal has been proved for these applications, their fracture toughness is still a serious cause of concern. Furthermore, their machineability is another serious issue. Work has been done on enhancing their fracture toughness and machineability by several means. The works of fracture toughness improvement include mainly the usage of reinforcing agents like fibers and whiskers. The fibers and whiskers used for this purpose are CFs, ceramic fibers, etc. Similarly, the machineability, more specifically wire EDM machineability, has been improved by means of conducting phases [1]. The conducting phases include again fibers, whiskers, etc.

The usage of reinforcing and conducting phases is a smart choice for almost eliminating the above mentioned two major flaws of ceramics. In this regard, the abundant work has been done. Although the usage of continuous fibers as a conductive reinforcing phase has been studied in depth, the matrix build up in continuous fiber preforms is a very challenging task. Several cycles of infiltration-pyrolysis and vapor deposition are needed for achieving required degree of densification. Alternately, hot-pressing route was explored as a rapid means for the fabrication of ceramic matrix composites. The hot-pressing route, mainly, use the sintering additives for binding the matrix and reinforcing phases. Although, some work has been done on short CF reinforced composites, the work on the usage of short fiber reinforced SiC composites is not much abundantly available. In last couple of years, however, short fiber reinforced composites have emerged as the commercially viable choice over continuous fiber reinforced composites, for various applications, due to several reasons [2–7].

The major challenge, however, in the usage of short CFs as reinforcement for such composites is their effective dispersion in matrix phase. Furthermore, machineability and fracture toughness along with morphological and mechanical performance of these ceramic composites greatly depend upon how the reinforcing phase is dispersed in the binder phase or more specifically in the ceramic matrix [8,9]. The properties gradually increase as the vol % of CFs increases [10]. Furthermore, due to low density of CFs, the net density of composites comes down. However, the air void

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content with increasing vol% of CFs becomes excessively high. It has been known that the air void content degrade the properties, mainly compressive properties of the composites [8,10,11]. This air void content is more prominent in CF reinforced cement composites. Furthermore, the increase vol% of CFs makes the handling or workability of composites troublesome [8,10]. Hence, judicious usage of CFs is foremost important. Thus the dispersion of CFs in continuous phase becomes very important.

In ceramic composites, air void influence is not highly prominent. However, porosity due to poor dispersion of CFs arises in ceramic matrix composites, mainly in those which are processed through hot-pressing. Furthermore, uniform dispersion is most important in achieving uniform wire EDM machineability. If CFs, which are conducting phase in ceramic matrix composites, are not uniformly disperse then the conductivity may not be uniform throughout. As a consequence, the wire may not move due to pocketing of conducting phase as a result of poor dispersion. Furthermore, the usage of CF reinforced composites at higher temperature without any oxidation resistance coating demand lower vol% of CFs. In this condition, this specific demand along with good wire EDM machineability can be met by the effective and efficient dispersion and distribution of CFs in continuous phase.

The dispersion of CFs in cement composites has been extensively studied [10,12]. Furthermore, several methods have been explored for checking the degree of CF dispersion in continuous phase. These include fresh mixture method, electrical resistance method, scanning electron microscope method and simulation experiment method. However, the best and versatile characterization of fibers' dispersion and distribution in powders is SEM micrographs based. This method gives actual dispersion [13].

The dispersion of CFs in various cement composites has been accomplished by the usage of dispersing agents, surfactants, foaming agents. Several types of dispersing agents like CMC, MC, HEC, etc., have been used. Furthermore, enormous work has been done on the usage of silica fumes (a fine particulate) as an admixture to enhance the dispersion of CFs in continuous phase.

Though some work on the processing of short CFs reinforced SiC matrix composites has been reported and further work is in progress, the specific work on the dispersion of CFs in SiC powder is not done. Furthermore, in the literature reported on short CF reinforced SiC composites, the dispersion is carried out with the help of dispersing agents like MC, CMC, HEC, etc., and defoamers like tributyl phosphate, etc [4,6,14,15]. The major issue with the usage of dispersing agents is the contamination in the CF reinforced SiC matrix. Additionally, the dispersing time of CFs in SiC matrix even with the help of these dispersing agents is quite high. The enhanced time of ball milling has resulted in the damage to CFs as reported by Hao et al. [16]. In the present scenario, on the processing of short CF reinforced SiC composites, it is the need of the hour to explore innovative and out of the box dispersing techniques along with processing methodology. On this line, as reported earlier [17], we developed a methodology to rapidly disperse CFs in SiC powder even without using any dispersing agent. Though we could disperse CFs up to 50 vol% excellently in SiC powder, the distribution for 30 and above vol% was found to be moderately good. In order to improve upon the distribution of CFs in SiC powder for higher vol % of CFs, we decided to explore the possibility of the same by tuning the ball milling parameters. To sort out the issue of distribution of CFs in SiC powder for higher loading the objectives, as briefed in the subsequent section, were set for the work.

1.1. Objectives

The main objective of this research work was to make the exfoliation-assisted dispersion of CFs in SiC powder commercially viable by enhancing the milling capacity without altering the dispersion and distribution or with enhanced degree of dispersion and distribution. On this line, how the milling parameters could affect the dispersion, distribution and APS, were to be studied. Furthermore, the effect of MT and BPR on exfoliation-assisted dispersion of short CFs in SiC powder was to be checked. Another objective was to explore the influence of SMPR on the dispersion of CFs in SiC powder and APS of powder mixtures.

2. Experimental procedures

2.1. Sample preparation

PAN based CFs (T-300, 3 K, Torayca Co, Japan, 3 mm length) and SiC powder (α-grade, Saint Gobain, Norway, 7.361 μm APS-volume weighted mean,) were taken for the milling experiments. Isopropyl alcohol (LR grade) was employed as a slurry media for the wet milling of SiC powder and CFs. It is to notworthy that both CFs and SiC powder were considered for BPR and SMPR calculations. The main characteristics of CFs, SiC powder and isopropyl alcohol were given elsewhere [1]. The microstructure of SiC powder is given in Fig. 1.

The continuous CFs from fiber spool were chopped into discrete length CFs using in-house designed and developed fiber milling equipment. The details of producing discrete length CFs were reported elsewhere [18]. The discrete CFs, thus obtained, were exfoliated. The microstructure of discrete and exfoliated CFs is given in Fig. 1. The exfoliation was followed by wet milling. The wet milling was carried out in isopropyl alcohol using planetary ball mill (Fritsch make, Pulversette P5 model). The bowls (SS with inner coating of silicon–nitride) of size Ø74.5 mm × 70 mm and the balls (Small ball of Ø10 mm and big ball of Ø20 mm) were used for this operation. The material of construction of balls was also silicon–nitride. The size of the balls was fixed as per previous work [17].

The powder mixtures were milled for different durations depending upon the parameter being optimized. However, milling cycle of 30 min with a soaking time of 5 min after each cycle and rapm of 120 were followed through out the milling operation. The milling cycle and rpm were used as per prious work [17]. The slurries of the samples were dried for 8 h at 90 °C @ 5 °C/min. After air drying powder mixtures were found to have certain agglomerates. To breakdown these agglomerates to finer scale, the powder mixtures were crushed using domestic mixer-grinder. The more details of these operations were described elsewhere [17].

2.2. Measurements and analytical methods

2.2.1. Visual inspection

The preliminary optimization of ball milling parameters like MT, SMPR, BPR, etc., was done through visual inspection of slurries. The fine tuning of these milling parameters was done through microstructural analysis. The degree of visual mixing of CFs in SiC powder was taken as the basis for visual inspection. The samples which show, visually, no sign of CFs accumulation, bundling, ball formation, etc. were only considered for further analysis through microstructure and APS.

2.2.2. Morphology

The morphology of the raw materials and powder mixtures were analyzed using Carl Zeiss make, SMT EVO 50 model, SEM and Carl Zeiss make, SIGMA HD model, FESEM, respectively. The SEM micrographs of un-exfoliated and exfoliated CFs, and SiC powder were taken after chopping and exfoliation, respectively. The FESEM micrographs of powder mixtures were taken after ball