PERGAMON

Carbon 39 (2001) 1119–1125

CARBON

Review article

Comparison of submicron-diameter carbon filaments and conventional carbon fibers as fillers in composite materials

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Received 10 September 2000; accepted 29 November 2000

Abstract

This review provides a comparison of submicron-diameter carbon filaments and conventional carbon fibers as fillers in composite materials. Carbon filaments (0.1 μm diameter, catalytically grown) are superior to conventional carbon fibers (discontinuous) as a filler in providing polymer–matrix and cement–matrix composites for electromagnetic performance, but are inferior to the fibers as a filler for composites for electrical and mechanical performance. Concerning the electromechanical behavior, the filaments are inferior to the fibers for cement–matrix composites, but are superior to the fibers for polymer–matrix composites.

Keywords: A. Carbon fibers; Carbon filaments; Carbon composites; D. Mechanical properties; Electrical (electronic) properties

1. Introduction

Carbon fibers made by pyrolysis from pitch and polymers [1–3] are widely used as fillers in composite materials, particularly lightweight polymer–matrix composites. A less common form of carbon is carbon filaments made catalytically from carbonaceous gases at 500–700°C [4,5]. A main difference between conventional carbon fibers made from pitch or polymer (typically of diameter ranging from 7 to 15 μm) and catalytically-grown carbon filaments (typically of diameter ranging from 0.01 to 0.2 μm) is in the diameter. In addition, conventional carbon fibers (referred to as fibers) can be in continuous and discontinuous forms, whereas catalytically-grown carbon filaments (referred to as filaments) are discontinuous only. Furthermore, a fiber does not have a hollow channel inside along its axis, whereas a filament does.

Fibers and filaments also differ in the microstructure of the carbon. Fibers have the carbon layers preferably oriented along the fiber axis, whereas filaments do not necessarily have this preferred orientation. A form of filaments which does not have this preferred orientation has the carbon layers at an angle to the fiber axis (resembling a fish bone). This is the form emphasized in this review, due to its wide availability in the USA. However, another form of filaments has tree-ring orientation of the carbon layers. The microstructure depends on the processing conditions during filament fabrication, but the correlation between microstructure and processing is beyond the scope of this paper.

Filaments are to be distinguished from carbon nanotubes, which are smaller (nm scale) in diameter and have the carbon layer(s) in the form of concentric cylinder(s) along the nanotube axis. For a single-wall nanotube, there is only one carbon layer. For a multi-wall nanotube, there are a few carbon layers. Both filaments and nanotubes have a hollow channel inside along the axis. Filaments are much lower in cost and wider in availability than nanotubes. Nanotubes can also be prepared by catalytic growth, though alternate methods include arc discharge and laser ablation.

Carbon filaments are also to be distinguished from vapor grown carbon fibers (VGCF), which are prepared by pyrolysis of carbonaceous gases to non-catalytically deposit carbon on catalytically grown submicron diameter carbon filaments at 950–1100°C [6–13]. The diameter of VGCF is typically larger than that of carbon filaments; it can be as large as a conventional carbon fiber.

Carbon fibers are mainly used in the form of composite materials. Therefore, evaluation of the performance of carbon filaments in comparison to carbon fibers as fillers in

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0008-6223/01/$ – see front matter
PII: S0008-6223(00)00314-6

composite materials is desirable. This paper is a review that provides this comparison in terms of the mechanical, electrical, electromagnetic and electromechanical behavior of the composite materials. The mechanical behavior pertains to structural applications. The electrical behavior pertains to electrical conduction applications. The electromagnetic behavior pertains to electromagnetic interference (EMI) shielding, microwave waveguides, electrostatic discharge protection, lightning protection, and lateral guidance in automatic highways. The electromechanical behavior pertains to strain/stress sensing, which is needed for smart structures, structural vibration control, traffic monitoring and weighing in motion.

2. Carbon filament description

The carbon filaments (lot ADNH-62) used were supplied by Applied Sciences, Inc. (Cedarville, OH). The filaments were made using methane as the primary source gas and an iron-containing catalyst. Hydrogen sulfide was added to the feedstock in small amounts to increase the filament yield. It has been reported that the sulfur addition causes the iron to melt and encourages filament growth by the vapor–liquid–solid process, as hydrocarbons adhere better to molten particles and carbon atoms may diffuse more rapidly through molten particles [14].

To assess the impact of crystallinity on the electrical performance of the carbon filaments, a filament sample was sent to St. Mary's Carbon Co. (St. Mary's, PA) for graphitization at 2600°C. The filaments were placed on a graphite sagger and packed on all sides in graphitized charcoal to a depth of ~1 inch. The graphitized charcoal was then surrounded by an additional layer of ungraphitized charcoal. The self generating atmosphere (produced by the volatilization of the ungraphitized charcoal) protected the load from oxidation by air. Current was passed through the graphite sagger at a rate of 8 kW/lb, thus heating the load to the graphitization temperature after ~32 h. The load was held at the graphitization temperature for 10–12 h. The power was disconnected and the filaments were allowed to cool to room temperature — a slow process requiring 3 to 4 days. X-ray diffraction showed that graphitization caused the 002 graphite peak to be much more intense and sharp, indicating increased crystallinity.

The basic properties of carbon filaments, as provided by Applied Sciences Inc., are listed in Table 1. The surface area in Table 1 was calculated, according to R.L. Alig of Applied Sciences Inc., by assuming that the fiber is a solid cylinder with a density of 2 g/cm^3. This density was used to obtain the filament volume fraction of composites.

A filament is actually a microtube. The inner hole diameter, from Applied Sciences' limited SEM photographs, varies from ~20 to 75 nm.

Table 1
Properties of carbon filaments

Diameter	0.16 μm
Surface area	12.5 m^2/g
Bulk density (compressed)	1620 cm^3/g
Surface treatment	Nitrogen groups
Sizing	None
SEM morphology	Entwined mass
Density	2 g/cm^3
Aspect ratio	50–200

3. Carbon filament composite processing

Due to the clingy morphology and small diameter of the carbon filaments, dispersion of the filaments in a composite requires more care than that of conventional short carbon fibers in a composite. A method of dispersing the carbon filaments in a thermoplastic matrix is slurry processing, which involves (i) dispersing the filaments in an isopropyl alcohol aqueous solution with the help of a trace amount of a dispersant, such as Triton X102 of Rohm and Haas Co. (Philadelphia, PA), (ii) mixing the slurry with thermoplastic powder at room temperature by using a kitchen blender, such that the concentration of isopropyl alcohol in the aqueous solution is adjusted so that the thermoplastic particles are suspended in the solution, (iii) draining the solution, (iv) drying at 120°C, and (v) hot pressing uniaxially above the glass transition temperature of the thermoplastic and at 1000 psi (6.9 MPa) for 0.5 h. The mixing in step (ii) causes very little filament breakage, so the aspect ratio of the filaments after mixing remains high (typically >1000) [15,16]. In this method, the thermoplastic must be in the form of fine particles (the finer, the better), due to the small diameter of the carbon filaments. As thermoplastics are mostly in the form of pellets rather than particles, the choice of thermoplastics is limited. In the case of a thermosetting resin such as epoxy, dispersion of the carbon filaments requires dilution of the resin with a solvent so as to lower the viscosity, and subsequent mixing of the filament–resin slurry by using a vigorous means, such as a blender. Due to the strong effect of the form of the matrix raw material on the dispersion of the carbon filaments, the properties (both mechanical and electromagnetic) of the composites significantly depend on the matrix material.

Conventional polymer processing methods such as extrusion and injection molding have been used for fabricating carbon filament polymer–matrix composites with thermoplastic matrices [17,18]. A high shear rate and a high draw ratio can cause some degree of filament alignment.

Catalytically grown carbon filaments tend to have a layer of polyaromatic hydrocarbons on their surface, due to the process in which the filaments are grown [19]. The hydrocarbon layer can be removed by cleansing with a

solvent, such as acetone and methylene chloride [19]. The removal of the hydrocarbon layer improves the bonding between carbon filaments and a thermoplastic matrix, as suggested by the fact that the volume electrical resistivity of the composite is much lower when the hydrocarbon layer on the filaments has been removed prior to incorporating the filaments in the composite [16,20].

The bond between carbon and a cement matrix is weak compared to that between carbon and a polymer matrix. Therefore, surface treatment of carbon for improving the bond with cement is particularly important. The treatment of catalytically grown carbon filaments (0.1 μm diameter) with ozone gas (0.3 vol.% in air, 160°C, 10 min) increases the tensile strength, modulus and ductility, and the compressive strength, modulus and ductility of cement pastes, relative to the values for pastes with the same volume fraction of untreated filaments [21]. Similar effects apply to the ozone treatment of carbon fibers [22], for which it has been shown that the ozone treatment improves the wetting by water, the degree of fiber dispersion in cement, and bond strength with cement, in addition to increasing the surface oxygen concentration [23,24].

4. Mechanical behavior

Due to the discontinuous nature, carbon filaments are far less effective than continuous carbon fibers as a reinforcement in composites. Due to their small diameter and the consequent large area of the interface between filaments and matrix in a composite, and due to their texture with the carbon layers at an angle from the filament axis, carbon filaments are not as effective as short carbon fibers (based on isotropic pitch) at the same volume fraction as a reinforcement, as shown for both thermoplastic [15,25] and cement matrices [26]. For example, in a thermoplastic matrix (slurry processing), carbon filaments at 19 vol.% give a tensile strength of 27 MPa [15], whereas carbon fibers (isotropic pitch based, 3000 μm long) at 20 vol.% give a tensile strength of 64 MPa [25]; in a cement paste matrix, carbon filaments at 0.5 vol.% give a tensile strength of 1.2 MPa, whereas carbon fibers (isotropic pitch based, 5 mm long) give a tensile strength of 1.7 MPa [26]. Although the filaments do not reinforce as well as the fibers, they still reinforce under tension. In a cement matrix, the tensile strength, modulus and ductility and the compressive modulus are all increased by the filaments (0.5 vol.%), but the compressive strength and ductility are decreased by the filaments [26]. In the case of polymer–matrix composites made by extrusion and injection molding, the tensile properties obtained with carbon filaments are comparable or inferior to those obtained with discontinuous PAN-based carbon fibers [27].

5. Electrical behavior

The DC electrical resistivity of polymer–matrix composites with polyethersulfone (PES) as the matrix and made by slurry processing is shown in Table 2 [15,21,28]. All the composites have some degree of preferred orientation of the filler in the plane of the resistivity measurement. For any of the electrically conducting fillers, the resistivity of the composite decreases monotonically with increasing filler volume fraction. The fillers are carbon filaments (0.1 μm diameter, not graphitized), carbon fibers (15 μm diameter, made from isotropic pitch, provided by Ashland Petroleum Co., Ashland, KY), nickel filaments (0.4 μm diameter, made by electroplating nickel on 0.1 μm diameter carbon filaments that have not been graphitized), nickel fibers (2 μm and 20 μm diameter) and aluminum flakes (1.2×1.0×0.03 mm). At about the same volume fraction (37–40%), carbon filaments give a composite of higher resistivity than nickel filaments, carbon fibers (400 μm diameter), nickel fibers (both 2 μm and 20 μm diameter) and aluminum flakes do. The lowest resistivity is provided by aluminum flakes. The second lowest resistivity is provided by nickel fibers (2 μm diameter). The third

Table 2
DC electrical resistivity of PES–matrix composites

Filler type	Filler vol.%	Resistivity (Ω cm)	Ref.
C fibers (100 μm long)	40	3.5	[15]
C fibers (200 μm long)	40	3.8×10^{-2}	[15]
C fibers (400 μm long)	40	1.1×10^{-2}	[15]
C filaments	25	6.2×10^{-2}	[21]
(0.1 μm diameter, not graphitized)	37	3.6×10^{-2}	[21]
Ni filaments	37	4.2×10^{-4}	[21]
(0.4 μm diameter)			
Ni fibers	37	1.4×10^{-4}	[28]
(2 μm diameter)			
Ni fibers	37	6.6×10^{-4}	[28]
(20 μm diameter)			
Al flakes	40	6.6×10^{-5}	[15]

Table 3
DC electrical resistivity of compacts of filaments and fibers

Material	Filaments or fiber vol.%	Resistivity (Ω cm)	Ref.
C filaments (0.1 μm diameter, not graphitized)	28	4.1×10^{-2}	[28]
C filaments (0.1 μm diameter, graphitized)	27	7.9×10^{-3}	[29]
	45	4.2×10^{-3}	
Ni filaments (0.4 μm diameter)	15	1.4×10^{-4}	[28]
Ni fibers (2 μm diameter)	15	9.5×10^{-5}	[28]
	24	3.6×10^{-5}	
Ni fibers (20 μm diameter)	30	8.2×10^{2}	[28]
	37	1.4×10^{-3}	

lowest resistivity is provided by nickel filaments (0.4 μm diameter). Hence, the carbon filaments fail to compete well with other fillers in providing composites of low electrical resistivity.

Table 3 shows the resistivity of compacts under pressure, which controls the volume fraction of the filaments or fibers in the compact [28,29]. The compacts are like composites without a matrix. The carbon filament compact exhibits higher resistivity than nickel filament and nickel fiber (2 μm diameter) compacts which have even lower volume fractions. Graphitization of the carbon filaments decreases the resistivity, but the value remains higher than those of nickel filament and nickel fiber (2 μm diameter) compacts of lower volume fractions. The absence of a matrix allows the carbon filaments to be in direct contact at their junctions. Nevertheless, at similar volume fractions, the carbon filament PES–matrix composite and carbon filament compact exhibit similar resistivity. Hence, the contact resistivity at the junction of filaments that are in direct contact is substantial.

The results in Tables 2 and 3 are consistent in showing that the carbon filaments are not as effective as some other fillers in providing composites of low resistivity.

In the case of carbon filament polymer–matrix composites made by extrusion and injection molding, the electrical resistivity is even lower than that of composites made by slurry processing [30].

6. Electromagnetic behavior

The electromagnetic behavior is relevant to electromagnetic interference (EMI) shielding, electromagnetic reflection and surface electrical conduction.

EMI shielding is in critical demand due to the interference of wireless (particularly radio frequency) devices with digital devices and the increasing sensitivity and importance of electronic devices. EMI shielding is one of the main applications of conventional short carbon fibers [31]. Due to the small diameter, carbon filaments (diameter 0.1 μm) are more effective at the same volume fraction in a composite than conventional short carbon fibers for EMI shielding, as shown for both thermoplastic [15,26] and cement [23,32] matrices. For example, in a thermoplastic matrix, carbon filaments at 19 vol.% give an EMI shielding effectiveness of 74 dB at 1 GHz [26], whereas carbon fibers (isotropic pitch based, 3000 μm long) at 20 vol.% give a shielding effectiveness of 46 dB at 1 GHz [15]. In a cement–matrix composite, fiber volume fractions are typically less than 1%. Carbon filaments at 0.54 vol.% in a cement paste give an effectiveness of 26 dB at 1.5 GHz [23], whereas carbon fibers (isotropic pitch based, 3 mm long) at 0.84 vol.% in a mortar give an effectiveness of 15 dB at 1.5 GHz [32]. These effectiveness measurements were made with the same fixture and about the same sample thickness. A low volume fraction of the filler is attractive for maintaining ductility or resilience in the polymer–matrix composite, as both ductility and resilience decrease with increasing filler volume fraction. Resilience is particularly important for EMI shielding gaskets and electric cable jackets. In addition, a low volume fraction of the filler reduces the material cost and improves the processability of the composites, whether polymer–matrix or cement–matrix composites.

The greater shielding effectiveness of the filaments compared to the fibers is because of the skin effect, i.e. the fact that high frequency electromagnetic radiation interacts with only the near surface region of an electrical conductor. However, carbon filaments are still not as effective as nickel fibers of diameter 2 μm at the same volume fraction, as shown for a thermoplastic matrix [21]. On the other hand, by coating a carbon filament with nickel by electroplating, a nickel filament (0.4 μm diameter) with a carbon core (0.1 μm diameter) is obtained [21,28]. The nickel filaments (0.4 μm diameter) are more effective than the nickel fibers (2 μm diameter) for shielding, due to their small diameter. At 1 GHz, a shielding effectiveness of 87 dB was attained by using only 7 vol.% nickel filaments in a thermoplastic matrix [21]. The shielding is almost all by reflection rather than absorption.

The high radio wave reflectivity of carbon filament (0.1 μm diameter) reinforced cement paste (at 1 GHz, 10 dB higher than plain cement paste) makes carbon filament concrete attractive for use in lateral guidance in automatic highways [33]. Automatic highways refer to highways which provide fully automated control of vehicles, so that safety and mobility are enhanced. In other words, a driver does not need to drive on an automatic highway, as the vehicle goes automatically, with both lateral control (steering to control position relative to the center of the traffic lane) and longitudinal control (speed and headway). Current technology uses magnetic sensors together with magnetic highway markings to provide lateral guidance, and uses radar to monitor the vehicle position relative to other vehicles in its lane for the purpose of longitudinal guidance. Cement paste containing 0.5 vol.% carbon filaments exhibits reflectivity at 1 GHz that is 29 dB higher than the transmissivity. Without the filaments, the reflectivity is 3–11 dB lower than the transmissivity.

The surface impedance of carbon filament composites, nickel filament composites and nickel fiber composites are low. In particular, at 1 GHz, the surface impedance is comparable to that of copper for a thermoplastic–matrix composite with 7 vol.% nickel filaments and a thermoplast–matrix composite with 13 vol.% nickel fibers (2 μm diameter) [26]. The surface impedance is higher for carbon filament composites than nickel filament composites or nickel fiber (2 μm diameter) composites at similar filler volume fractions [26]. Although carbon filaments have a lower density than nickel filaments, a thermoplastic–matrix composite with 7 vol.% nickel filaments has the same specific surface conductance (surface conductance divided by the density, where conductance is the reciprocal of impedance) as one with 19 vol.% carbon filaments [26]. The low surface impedance is valuable for applications related to electrostatic discharge protection and microwave waveguides.

7. Electromechanical behavior

The electromechanical behavior relates to the effect of strain or stress on the electrical resistivity. This phenomenon is known as piezoresistivity. It is relevant to strain/stress sensors.

Strain sensors refer to sensors of strain, which relates to stress. The strain sensed includes reversible and irreversible strains. Due to the advent of smart structures, strain sensors are increasingly needed for structural vibration control and in situ structural health monitoring. Composites containing conventional short carbon fibers have their volume electrical resistivity change reversibly upon reversible strain, thus allowing the composites to serve as strain sensors. In the case of a composite with a ductile matrix (such as a polymer matrix), this phenomenon is due to the change in the distance between adjacent fibers in the composite [34]. Tension causes this distance to increase, thereby increasing the resistivity; compression causes this distance to decrease, thereby decreasing the resistivity. In the case of a composite with an elastomer matrix (such as a silicone matrix), the phenomenon is different in both direction and origin; the resistivity decreases upon tension, as observed for a silicone–matrix composite with 0.4-μm-diameter nickel filaments (with a 0.1-μm-diameter carbon filament core in each nickel filament [21]) [35]. This reverse piezoresistivity effect is probably due to the increase in filament alignment upon tension. In the case of a composite with a brittle matrix (such as a cement matrix), the phenomenon is not reverse but is yet different in origin; it is due to the slight (<1 μm) pull-out of the fiber (short) bridging a crack as the crack opens and the consequent increase in the contact electrical resistivity of the fiber–matrix interface [36–38]. Tension causes a crack to open, thereby increasing the resistivity; compression causes a crack to close, thereby decreasing the resistivity.

The use of carbon filaments (0.1 μm diameter) in place of conventional short carbon fibers (based on isotropic pitch) in a polymer–matrix composite improves the reproducibility and linearity of the piezoresistivity effect (not reverse) [39]. This is because of the small diameter of the filaments, which results in (i) a large number of filaments per unit volume of the composite, (ii) reduced tendency for the filaments to buckle upon compression of the composite, and (iii) reduced tendency for the matrix at the junction of adjacent filaments to be damaged. Furthermore, the use of the filaments enhances the tendency for the reverse piezoresistivity effect [40].

The use of carbon filaments (0.1 μm diameter) in place of conventional short carbon fibers (based on isotropic pitch) in a cement–matrix composite results in increased noise in the electromechanical effect [41]. This is because of the bent morphology and large aspect ratio of the filaments, which hinder the pull-out of filaments. Thus, carbon filaments are not attractive for cement–matrix composite strain sensors.

8. Conclusion

Carbon filaments (0.1 μm diameter, catalytically grown) are superior to conventional carbon fibers (discontinuous) as a filler in providing polymer–matrix and cement–matrix composites for EMI shielding, electromagnetic reflection and surface electrical conduction. The superiority of the filaments is further enhanced by coating the filaments with nickel. However, the filaments are inferior to the fibers ($\geq$400 μm long) as a filler in providing polymer–matrix composites for DC electrical conduction, or in providing polymer–matrix and cement–matrix composites for mechanical performance. In relation to the electromechanical behavior, the filaments are much inferior to the fibers for cement–matrix composites, but are superior (in terms of

the reproducibility and linearity of the piezoresistivity effect) to the fibers for polymer–matrix composites.

Acknowledgements

This work was supported in part by National Science Foundation, USA, and in part by Defense Advanced Research Projects Agency, USA

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