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PITCH BASED SHORT CARBON FIBER

SIN-SHONG LIN
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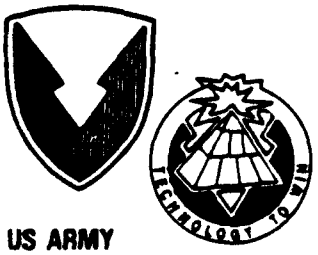

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ABSTRACT

Short carbon fiber manufactured from coal tar pitch by Osaka Gas Co. is examined by chemical composition analysis, X-ray powder diffraction, optical microscope, and electron spectroscopic techniques. The present analytical results are compared with the data obtainable from other sources. Owing to the low cost of the short fiber, it is recommended that the fiber could be used for a wide variety of reinforcement applications such as, cement/concrete mixtures, polymer composites, and high temperature materials.

CONTENTS

INTRODUCTION

The short carbon fiber DONACARBO S210 manufactured by Osaka Gas Co. was derived from coal tar obtainable from the decomposition of natural gas. The processing technology is claimed to be novel and economical and that it would alter future outlook of competitive worldwide carbon fiber industries. The carbon fibers in the forms of short fibers and continuous filaments have been produced from coal tar for the first time.

This novel processing includes the mechanical separation of mesophase microbeads of three to 30 micron diameters from crude coal tar during three heat treatment stages. The mesophases obtained are then subjected to solvent extraction, hydrogenation, and polymerization to yield isotropic and anisotropic pitches suitable for melt spinning. The short fiber is fabricated from isotropic pitch by the rotary gas jet method, and the process yields a higher quality fiber as compared to other melt spinning methods. The most important feature is that this process is highly cost effective.

Specifically, the short carbon fiber derived from the coal tar precursor by high temperature spinning process has several unique properties claimed by the manufacturer. The short fibers produced by this process are curled lengthwise which might provide a better intimate contact among fiber strands in the reinforced composites. Thus, strength, electric conductance, magnetic shielding, thermal insulation, and sound damping are expected to improve considerably. Potential U.S. Army applications include lightweight portable bridges, missile casings, and many components of troop support equipment. The special features, uncommon to the well-known short carbon fiber derived from melt spinning by KUREHA Co., are listed as follows:

- Fibers are curved and are interwoven.
- High electric and thermal conductivities.
- Fiber cross section is not circular and varies from 8 μm to 16 μm.
- Tensile strength 0.7-1 GPa, modulus 30 GPa to 40 GPa and elongation 2%.
- Good corrosion resistance.
- Carbon content <95%.
- Good compatibility with polymer resin and good molding characteristics.
- Low cost.

The fibers are available in the forms of felt, paper, chopped, milled, and yarn. There are many applications of this short fiber such as high temperature insulation, structural resin with fiber impregnation, fiber reinforced concrete, electric shield, and corrosion resistance materials. The studies reported here comes from the "as grown" fiber obtained through the AMC STCFE Office, Yokota AFB, Japan.

The unique characteristics of this short fiber such as interfacial adhesion, microstructure, fiber strand orientation, and surface morphology have been extensively examined. The analytical techniques available at the U.S. Army Materials Technology Laboratory (MTL) such as chemical composition analysis, wide angle X-ray diffraction, optical microscopy, scanning electron microscopy (SEM), and Auger/photo electron spectroscopies (AES/XPS), were used in the present investigation.

CHEMICAL AND INSTRUMENTAL ANALYSES

Chemical Composition Analysis

The compositional analysis of the short fiber was performed by Galbraith Laboratories, Inc., Knoxville, TN. A small portion of the short fiber sample was enclosed in a sealed plastic envelop and was mailed for chemical analysis. The assay is made twice by atomic absorption spectroscopy using argon gas as a carrier. The result is tabulated in Table 1 below:

Table 1. CHEMICAL CONSTITUENTS OF SHORT FIBER

	Carbon*	Oxygen	Hydrogen	Nitrogen	Sulfur	Silicon
Run 1 (Wt%)	86.64	5.90	0.82	0.89	<0.5	<0.4
Run 2 (Wt%)	86.93	5.62	0.96	0.87	<0.5	<0.4
Average (Wt%)	86.73	5.76	0.89	0.88	-	-
Average (At%)	84.6	4.2	10.4	0.8	-	-

*Manufacturer claimed to have at least 95%.

In the last row of the table the chemical composition based upon the atomic percent is shown where high concentrations of hydrogen and oxygen exist. The high hydrogen content certainly indicated that a large fraction of polycyclic aromatic compounds (about 10%) still remained in the fiber. Thus, carbonization is believed to be incomplete after the fiber melt spinning process. Probably, either time or temperature is not sufficient for a complete transformation from coal tar pitch to carbon. A large abundance of oxygen present in the sample is a typical characteristic of low cost fibers derived from the coal tar precursor. Many hetro cyclic rings containing oxygen together with various carbon oxygen functional groups might be present as major structure components. Since few metallic elements are found as impurities in the analysis, the majority of oxygen is believed to be in the form of the carbon oxygen bonds. The carbon content (>95%) claimed by the manufacturer was not substantiated in this investigation.

X-Ray Powder Diffraction

The characterization by X-ray diffraction analysis was made using Norelco X-ray diffractometer and power supply units manufactured by Phillip Electronic Instrument Co. The fiber was first ground into powder by a SPEX miller and mounted on a standard specimen holder with radiation of a 1/2" x 1" area. The powder diffraction pattern from 4° to 60° was taken on a strip chart recorder using Cu-Kα X-ray radiation. The diffraction pattern was then transferred to an IBM XT personal computer using a graph plotter for digitalization. The computation of crystallographic data was then performed by the computer.

The crystallographic parameters computed are listed in Table 2. The D-002 spacing and the A-axis length are obtained from the peak positions of the diffractions 002 and 100. The two most important parameters, crystal sizes L_a and L_c, are calculated from the widths of the diffraction peaks 100 and 002 by the Scherrer Equation. In addition, the fraction of peak areas are obtained from the integration of all diffraction peaks respective to the total diffraction area.

In addition to the short fiber, the data from the continuous filament tows such as F-series made by the same manufacturer, Osaka Gas Co., from anisotropic pitch derived from coal tar, polyacrylonitrile (PAN) based fiber represented by TOHO RAYON IM600, and graphite are listed for comparison. For the unit cell parameters, the F500 fibers are found to have an identical unit cell dimension (columns 2 and 3) to that of graphite, while the F180 have fibers to that of the PAN-based fiber. The short fiber unit cell is nowhere near those of graphite or the fibers that come from anisotropic pitch. Moreover, the size of the short fiber crystallite (indicated by L_a and L_c in columns) is the smallest. The crystallite sizes of F180 are slightly larger than that of the Pan based one, and those of F500 are much larger. However, the sizes of the highly crystallized F500 fibers are still small as compared to graphite which is in the range of 1 μm (= 10000Å). The crystalline fraction (last column) of the coal tar fibers are between PAN and graphite. The crystalline fractions of F500 are more than 0.85 while that of the short fiber is the lowest.

Table 2. X-RAY ANALYSIS OF CARBON FIBERS DERIVED FROM COAL TAR (MANUFACTURED BY OSAKA GAS CO., JAPAN)

	D-002 Spacing (± 0.02 Å)	A-Axis Length (Å)	L_a (Å)	L_c (Å)	Peak Fraction (%)
DONACARBO					
S210-1	3.74	2.46	35.3	11.0	0.257
S210-2	3.69	2.43	29.9	11.4	0.269
DONACARBO					
F500-1	3.43	2.45	230	169	0.895
F500-2	3.44	2.45	160	274	0.858
F500-3	3.42	2.45	210	203	0.838
F500-4	3.39	2.44	140	402	0.886
F500-5	3.45	2.45	138	-	0.828
F180-1	3.51	2.43	62	34	0.634
F180-2	3.53	2.43	54	27	0.665
F180-3	3.54	2.44	65	28	0.588
TOHO IM600					
PAN Fiber	3.54	2.44	48	20	0.467
Graphite	3.42	2.46	~10000	~10000	1.0

A typical X-ray powder diffraction pattern of S210 together with Graphon (highly graphitized carbon black) and coke is shown on Figure 1. Crystallized carbon, such as graphite and Graphon, has a diffraction pattern consisting of well-defined peaks. On the contrary, highly disoriented carbon, such as coke, shows a poorly defined spectrum. The diffraction pattern of the short fiber is much closer to that of amorphous carbon. The 002 peak is significantly widened indicating that the structure is composed of smaller crystallites. The peak shifting toward low diffraction angles indicates that the interlayer distance between stacking hexagonal planes becomes large due to disorder and distorted arrays of the deformed carbon layers.

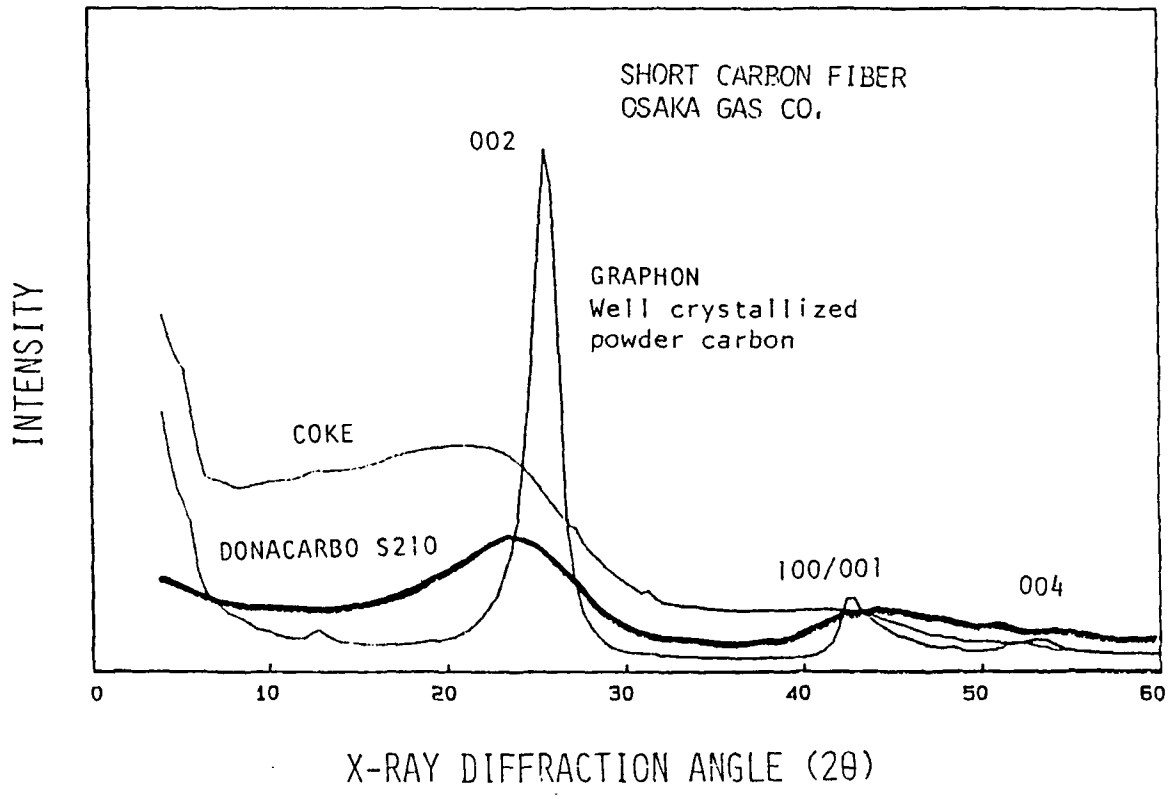


Figure 1. X-ray powder diffraction pattern of short fiber. The diffraction patterns of the short fiber, Graphon and coke are compared. Graphon is highly crystalized carbon and coke is amorphous carbon. The pattern of the short fiber is somewhere between these two.

Optical Spectroscopy

The appearance of the short fiber at low magnification by optical microscopy is shown in Figure 2a through 2c (magnified 25X, 50X, and 140X, respectively). The fibers have various widths ranging from 6 μm to 18 μm and are always curled along the length. It is important to note that each strand of the fiber is entangled with other strands at several crossings. Thus, the intimate contact could establish to enhance electric as well as thermal conductivities. Moreover, a high strength reinforced composite is possible due to extensive entanglement and interlacing.

In Figure 2, a general view of a short fiber bundle is shown. All fibers are randomly oriented and intersect each other at several locations. A single strand of fiber has a length from a few mm to several cm with average diameter approximately 10 μm. Some filament widths are larger than 15 μm. Various sizes of filament strands are clearly shown on Figure 2b. Generally, most of the fiber surfaces appear smooth under low magnification, but some segments are rugged terrains containing many intrusions and pits. In Figure 2c, different widths of the fibers are quite noticeable. These rough terrains presumably contain a large amount of foreign materials such as silicon or oxygen impurities, carbon particles, and high molecular weight insoluble hydrocarbons.

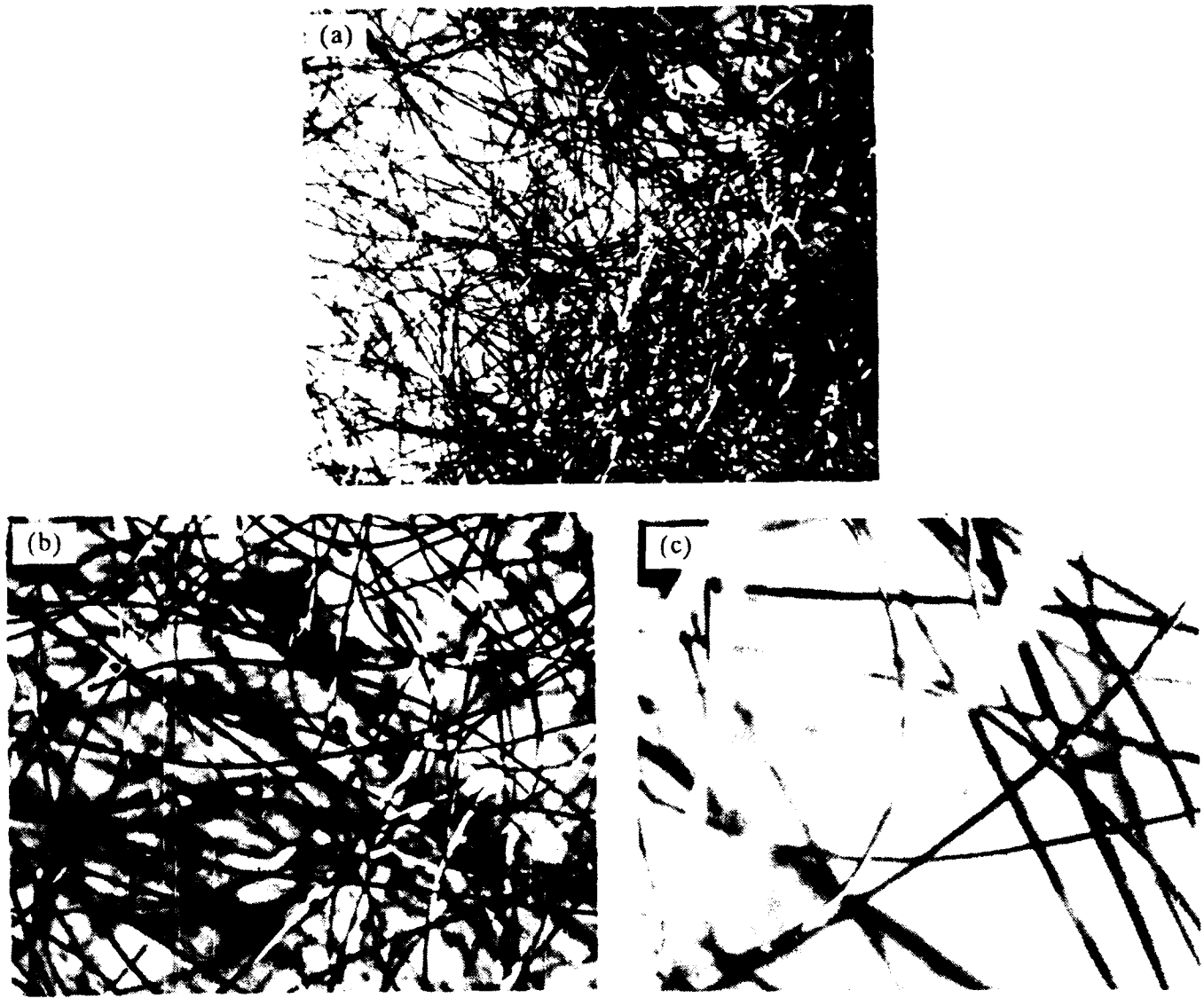


Figure 2. Optical micrographs of short fiber. The short fiber is manufactured by Osaka Gas Co. designated as DONACARBO S210. The photographs are taken at magnifications of (a) 25X, frame width 4 mm, (b) 50X, frame width 2 mm, and (c) 140X, frame width 0.7 mm. A wide variety of fiber lengths widths, and shapes are shown. The widths vary from 6 μm to 15 μm and lengths from a few mm to several cm long. Due to intimate contacts provided by curled filaments, this short fiber is claimed to possess high electric and thermal conductivities. A high strength reinforced composite with good electric and magnetic shield properties could be fabricated from the incorporation of this short fiber.

Scanning Electron Microscopy

The SEM examination reveals many detailed features of the fiber surface, as shown in Figure 3. The surface is generally characterized by longitudinal grooves and striations with numerous surface irregularities such as impurity intrusions, pits, voids, specks, and gains (see Figures 3a and 3b). Moreover, the filaments appear to have different shades along the fiber length suggesting that they are consisted of different and heterogeneous coal tar components. A large number of particles attached to the surfaces are visible and are probably derived from contaminations during the fabrication process. As shown in Figures 3c and 3d, fractured cross section surfaces are rather featureless indicating a poorly developed crystallinity. The cross

sections are not circular and a few ridges are oriented randomly at the darker center region in contrast to the skin portion where a slightly better crystal orientation is found. In many areas, debris left from the preparation are attached on the cross-sectional surfaces.

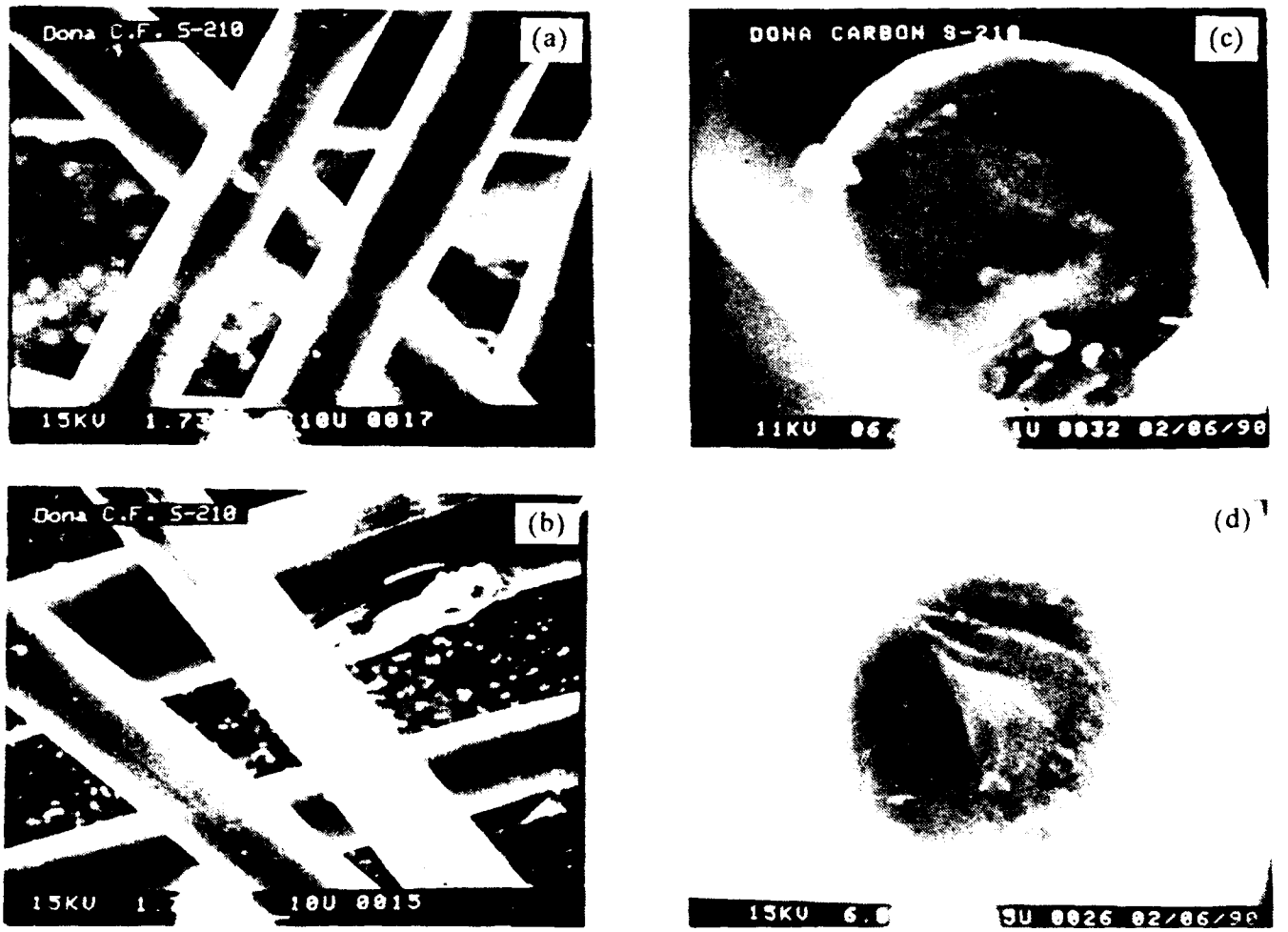


Figure 3. SEM micrographs of short fiber DONACARBO S210. (a) Surface features of the short fiber. Fiber surface is characterized by longitudinal grooves and striations with many irregularities: pits, voids, specks, and gains. The surface appears to have different shades, (b) ibid, (c) Fractured fiber cross sections. The fractured surfaces of fiber cross section is rather featureless indicating a poorly developed crystallinity, and (d) ibid.

Scanning Auger Microscopic Analysis

The information concerning the surface composition of the short fiber was obtained by Auger electron spectroscopy. The scanning auger microscopic (SAM) instrument XSAM800 manufactured by Kratos Analytical Co. was used throughout the present investigation. The spatial resolution of the electron beam was about 0.2 μm at 5 keV electron energy, with the electron current set in the range of 15 nA to 20 nA. The auger image was stored on the hard disk of the SMS 1000 minicomputer in situ. During imaging of carbon fiber surfaces, carbon in addition to oxygen was usually taken as a reference image. The data after acquisition were then manipulated to produce a two-dimensional display of surface oxygen. The scanning capability of the present instrument also provides two-dimensional images of the fiber surface morphology. The secondary electron detector (SED) produces the surface images similar to SEM photos except at a lower resolution.

Large segments of the short fiber surfaces are smooth as indicated in the SEM photos (see Figures 3a and 3b); however, some show many large irregularities, as shown in the SED images of Figures 4a and 4b. In Figure 4a, four strands of the short fiber intersect at the same crossing; two of the fibers have a diameter twice that of the others. Moreover, there is a large elongated pit several diameters long situated on the surface of the smaller one. The SAM analysis revealed that the pit surface is full of impurities such as oxygen, nitrogen, silicon, and sulfur in addition to the major component, carbon. Many defects and intrusions are lined along the fiber length in Figure 4b. Due to the nature of the low cost precursor by which the fiber is fabricated, these elements are integral parts of the fiber composition.

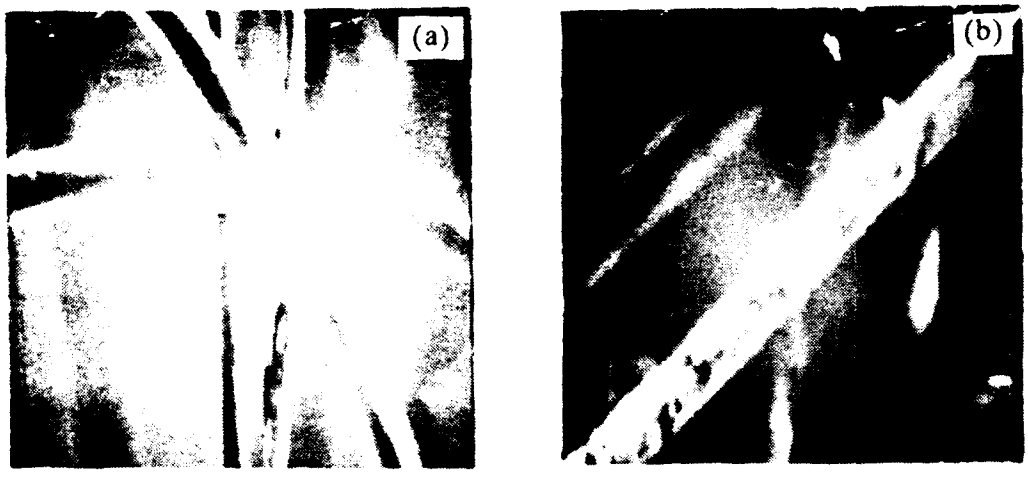


Figure 4. Scanning electron detector images of short fiber. (a) four filaments with varying sizes and surface morphologies. A large pit or defect is found on a small filament, and (b) a single strand of filament with many inclusions and defects.

A typical Auger spectrum of the short fiber surface is shown in Figure 5. Carbon and oxygen are the major elements existing together with minor elements such as nitrogen, sulfur, and silicon (not visible). Oxygen is believed to be incorporated as carbon-oxygen bonds such as ether, ester, or carbon oxygen functional groups (hydroxyl, carbonyl, and carboxyl), but it also could exist as silicon oxide impurities. Nitrogen and sulfur might be parts of carbon hetero ring structures. The surface elemental concentrations of this small spot about 0.2 μm x 0.2 μm in area calculated from peak areas of the spectrum are tabulated in Table 3.

Table 3. SURFACE COMPOSITION OF SHORT FIBER

Element	Position (eV)	Width (eV)	Area	Sensitivity factor	Atomic Mass	Atomic %	Weight %
Carbon	258.5	43.95	640612	0.14	12.01	98.30	97.31
Oxygen	499.0	9.34	26953	0.45	16.00	1.29	1.71
Nitrogen	381.0	3.5	4639	0.42	14.00	0.42	0.48

The oxygen distributions of the fiber surface are shown in Figure 6. The SAM mapping reveals that oxygen is distributed evenly throughout the surfaces except in these segments where a large amount of impurities existed. It is interesting to note that high oxygen concentrations are located along grooves and striations of the fiber in Figure 6a. Also, oxygen

depletion is observed in some fiber segments, as shown in Figure 6b. The oxygen rich sites are scattered in a random fashion across the fiber surface. The abundance, as well as the uniform distribution, of oxygen suggested that the fiber has reasonably good adhesion to polymer matrix.

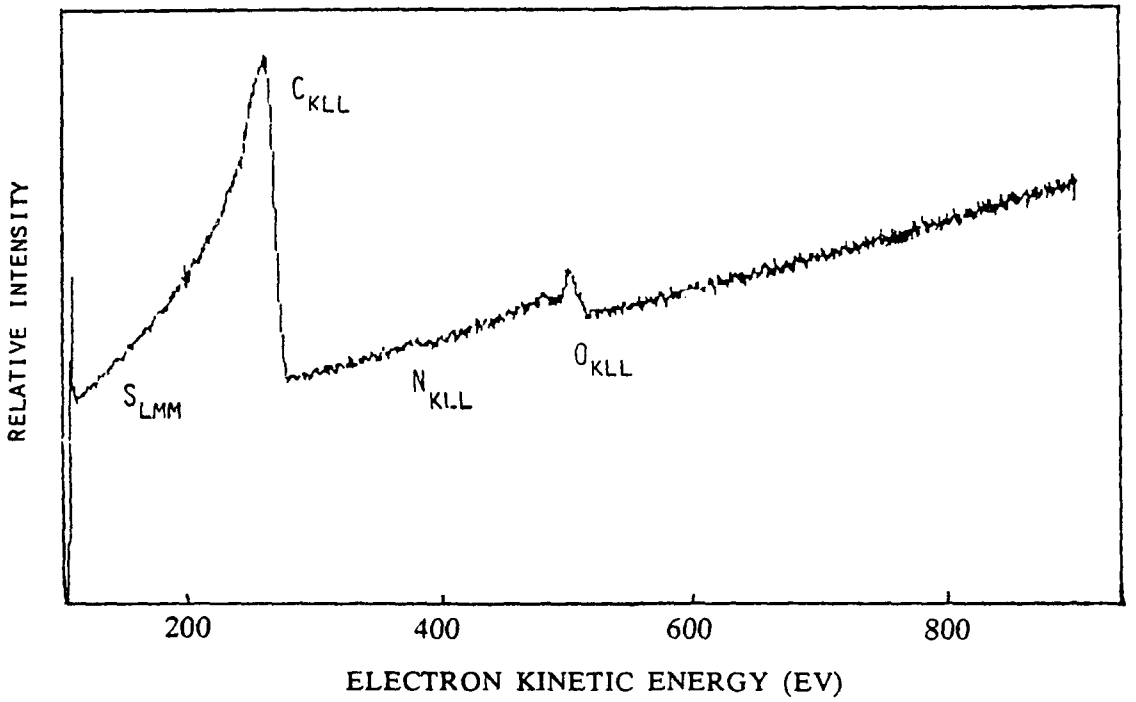
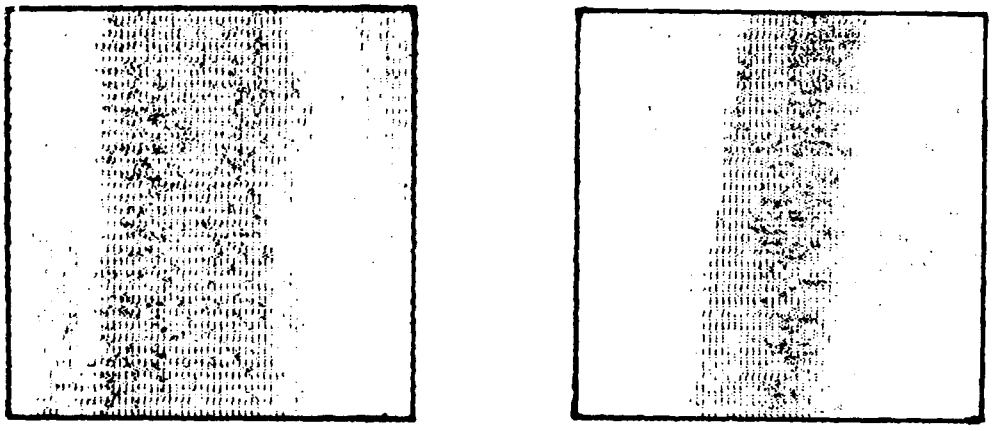


Figure 5. Typical auger electron spectrum of short fiber. Spectrum OFS001, oxygen, nitrogen, and sulfur are present together with carbon.



(a) Frame width 20 μm

(b) Frame width at 35 μm

Figure 6. Oxygen distribution of SAM images. (a) high oxygen concentrations at fiber striations and grooves, and (b) Aggregation of oxygen rich sites and oxygen difficient spots in some segments.

In general, the surface composition of the short fiber contained large amount of impurities such as oxygen, nitrogen, silicon, and sulfur in addition to carbon. The surface distribution of the second major abundant element, oxygen, is quite even and that of the minor elements, such as sulfur and silicon, is spotty. The presence of abundant surface oxygen and the rugged surface terrains suggest that the fiber adheres well to the matrix.

Interlaminar Shear Strength

The specimens for the purpose of evaluating the short fiber interlaminar shear strength were prepared from a resin containing 60 parts of Epoxy 828 and 40 parts of Epon V-40 curing agent. Due to poor dispersion of the present fiber in the epoxy matrix, the three point shear test according to ASTM D 790 might not yield a reliable result. Owing to the limited funding and manpower, the present tedious measurement is temporarily halted until further funding is available.

CONCLUSIONS

The physical and chemical characteristics of the short carbon fiber made by Osaka Gas Co. designated as DONACARBO S210 were evaluated by chemical compositional analysis, X-ray powder diffraction, optical microscopy, SEM, and AES for chemical composition, crystal structure, fiber geometry, fiber morphology, and surface composition. The information gathered through the present studies were compared with the data available from other fibers. The results have been discussed in the preceding analytical paragraphs.

The important features associated with the short fiber obtained from the present evaluations are:

- Fiber strands up to 2 cm long are curled and are interwoven.
  -- which could provide high thermal and electric conductances.
- Fiber cross sections are not round and the widths vary from 6 μm to 18 μm.
  -- which indicates that it came from a simple spinning production.
- Fiber surfaces are dotted with intrusion, extrusion, and impurities.
  -- which suggests that it is derived from an economical processing technique.
- Tensile strength 0.7-1 GPa, modulus 30 GPa to 40 GPa and elongation 2%.
  -- which might imply a good quality low cost fiber.
- Fiber structure consists of small carbon grains about 10 Å x 30 Å.
  -- not a highly graphitized structure.
- Fiber composition suggests that the fiber is not well annealed at high temperature.
- Fiber adhesion seems to be reasonably good based upon the oxygen content and the oxygen distribution. Good adhesion is an essential factor for the application of this short fiber to reinforced polymer/concrete composites.

This short fiber is a unique material made in Japan and not available elsewhere. Many features such as good tensile strength, good corrosion resistance, medium-high thermal and electric conductances, and low cost are required for a wide variety of applications including high temperature insulation, structural resin with fiber impregnation, fiber-reinforced concrete, electric shield, and corrosion-resistance materials.

RECOMMENDATION

This short fiber has been widely accepted in Japan for uses in high temperature insulation, heat and chemical resistance valves, and special corrosion filter materials. Furthermore, it has a potential application in low cost reinforced resin matrix and concretes. The present study has revealed many superior characteristics of the short fiber. Further exploitation is needed to illustrate detailed adhesive behavior and compatibility between the short fiber and polymer matrix. The essential criteria required for an appropriate selection of polymer matrix or concrete matrix for various reinforced composites should be carefully studied from the properties of this short fiber.

Many potential uses for this short fiber exist. The areas of the U.S. Army's immediate material application are:

- Reinforced mortar (or dry mixed mortar) for electro-magnetic interference shielding, high thermal conducting structures, and lightweight material for building construction.
- The short fiber wetted with 5% epoxy resin in a pellet form could be used in the fabrication of various polymer composites. The process is very cost effective. The applications are space heating panel, magnetic, electrostatic shieldings, and acoustic absorbents.
- The efficient preparation of carbon/carbon composites by resin impregnation could be made for vehicle brakes, high temperature gasket, fuel cell electrode, and high temperature refractory casting molds.

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Pitch based carbon fiber
Analyses

Technical Report MTL TR 91-51, December 1991, 13 pp-illus-tables, D/A Project: D650 #90111 FY91

Short carbon fiber manufactured from coal tar pitch by Osaka Gas Co. is examined by chemical composition analysis, X-ray powder diffraction, optical microscope, and electron spectroscopic techniques. The present analytical results are compared with the data obtainable from other sources. Owing to the low cost of the short fiber, it is recommended that the fiber could be used for a wide variety of reinforcement applications such as, cement/concrete mixtures, polymer composites, and high temperature materials.

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Watertown, Massachusetts 02172-0001
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