Award Accounts

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Preparation and Characterization of Carbon Microcoils (CMCs)

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Carbon micro-coils (CMCs) with 3D-helical/spiral structures and coil diameters on the order of micrometer were obtained by metal-catalyzed pyrolysis of acetylene at 700–800 °C. The preparation conditions, morphology, growth mechanism, microstructure, and some properties of the CMCs were examined in detail. Two pieces of vapor grown carbon fibers grown from a metal catalyst grain curl and entwine each other due to the anisotropic catalytic activity between different crystal faces of the catalyst grain and thus form double-helix CMCs. CMCs have an almost amorphous structure but could be graphitized by high-temperature heat treatment with full preservation of the coiling morphology. The CMCs could effectively generate inductive electromotive force, inductive current and thus Joul's heat under microwave irradiation. The CMCs/elastic polymer composite elements showed high tactile sensing properties which are comparable to that of human skin. The CMCs are a possible candidate for electromagnetic wave absorbers, remote heating materials, visualization elements of microwaves, tactile sensor elements, micro-antennae, chiral catalysts, bio-activators or bio-deactivators, energy converters, etc.

1. Introduction

1.1 Research Background. In the cosmos and in nature, maelstrom, spiral, helical, or coil structures, such as a whirled air (typhoon), whirled seawater, vine plants, shellfish, proteins, DNA, etc., are commonly observed. The 3D-helical/spiral forms are also commonly observed in artificial water screws, spiral staircases, retaining screws, bolts/nuts, screw dislocations in solids, etc. Furthermore, electromagnetic waves, musical scales, spirits or minds, economic cycles, etc. also move with the helical/spiral motion patterns. Recently, electron spins were found to have a spiral arrangement. In other words, 3D-helical/spiral structures are the fundamental structure of many objects and phenomena, in science and human livings from the cosmos scale to atomic scale. The helical morphology of DNA and proteins plays essential and critical functional roles in living bodies. Recently, the double-helix-shaped DNA, single-helix-shaped proteins, and chiral carbon nanotubes have attracted lots of interest in biotechnology and nanotechnology.[1–3] However, materials with 3D-helical/spiral structures with micrometer to nanometer dimensions are not commercially available.

1.2 Research History. In 1953, Davis et al.[4,5] first reported the vapor growth of thin carbon fibers twisted together in the form of a rope. The observation of the growth of helical/spiral-coiled carbon nanofibers (carbon coils) using a CVD process have been occasionally reported by many carbon filament researchers. However, before 1990, the carbon coils grown by the CVD process were extremely accidental, and the reproducibility was very poor. The production was basically ignored

because carbon fibers with helical/spiral-forms were considered to be the anomalous and unpreferable deposits. In 1989, Motojima et al.[6,7] found that very regularly coiled silicon nitride (Si_3N_4) fibers could be obtained by the CVD process. In 1990, Motojima et al.[8] found that regular-coiled carbon fibers (carbon microcoils) could be obtained with high reproducibility by the catalytic pyrolysis of acetylene containing a small amount of sulfur or phosphorus impurities over transitional-metal catalysts and then extensively examined the preparation conditions, morphology, growth mechanism, and some properties in detail. Motojima et al. named "cosmomimetic carbon microcoil (CMC)" for these carbon microcoils after "biomemetic."[1] Since 2000, the interest in the carbon microcoils has dramatically increased, and carbon coils with diverse morphologies have been reported by many researchers. Among various preparation processes, CVD has the most potential process for the large-scale preparation of the pure carbon coils. Thus, the preparation of carbon coils has been mainly carried out using various CVD reaction systems using Fe-group metal catalysts and acetylene or ethylene carbon sources.

1.3 Basic Research Concept. All of the things in the cosmos have common structures of maelstrom, spiral, helical, or coiling patterns. In other words, the 3D-helical/spiral structures are the fundamental structure of many natural objects and phenomena, including science and human livings, from the cosmos scale to atomic scale. The helical morphology of DNA and proteins has essential and critical functions in living bodies. Considering these facts, CMCs with 3D-helical/spiral micro structure of micrometer–nanometer dimensions should have novel functionalities which can not be obtained from con-

ventional materials. Actually, CMCs have been shown to have many novel functionalities and many potential applications, such as tunable microdevices/sensors, electromagnetic wave absorbers, energy changing materials, hydrogen absorbers, chiral catalysts, activators or deactivators of organisms, etc. In addition to their potential applications, the growth mechanism and microstructure are very interesting from a scientific point of view. A comprehensive review on CMCs was recently published by Motojima et al.[9–13]

1.4 Research Aim and Establishments.   Emphasis has been placed on the development of a series of novel 3D-helical/spiral materials, mainly CMCs. CMCs and various ceramic microcoils/microtubes were first prepared, and a mass production process was developed. The chemical, mechanical, electrical, electromagnetic, magnetic, biological properties of the CMCs were examined in detail, and many novel properties were discovered. Furthermore, various interesting and novel microstructures and growth mechanisms of 3D-helical/spiral patterns were developed, and possible applications were considered. The main research achievements on the preparation and characterization of the CMCs are described as the follows.

2. Experimental

CMCs were obtained by catalytic pyrolysis of hydrocarbons containing a small amount of sulfur or phosphorus compound impurities at 700–800 °C. As a carbon source, acetylene is the most effective for obtaining the CMCs with high purity and high yield. The presence of catalyst is indispensable for the growth of the CMCs as in the case of vapor grown carbon fibers (VGCF). As a catalyst, Ni fine powder (0.05–50 μm diam.) or Fe–Ni-based alloys supported on fine ceramic powders were the most effective and convenient catalyst, and these catalysts were mainly used in this work. A gas mixture of $C_2H_2 + H_2 + N_2 +$ impuirty was vertically introduced into horizontal reaction tubes (quartz, 30–100 mm i.d.) and exhausted through lower gas outlets. The standard reaction conditions used are as follows: reaction temperature = 750–800 °C, gas flow rate of acetylene = 150–200 sccm, impurity (thiophene) content in total gases = 0.6–0.8 vol %, gas flow ratio $(H_2/C_2H_2) = 3$–4.

CMCs can absorb very effectively an electromagnetic wave as will be shown later. Using a conventional electric heater, electromagnetic wave (28–33 mG at substrate surface) was irradiated from the electric heater into the growing atmosphere of the CMCs. It was observed that the outer electromagnetic wave (referred to as "EM wave" hereafter), emitted from the outer electric heater, and/or bias voltage strongly influenced on the growth and morphology of the CMCs. The coil yield that was obtained with irradiation of EM wave was 1.6–2.3 times higher than that without irradiation in which gas burner heater was used. Furthermore, the yield when a bias voltage (AC and DC) was applied to the substrate was 2.2–2.3 times higher than that without bias voltage, and the coil yield increased with increasing bias voltage. The coil yield also increased by applying ultrasonic wave (15–45 KHz) to the reaction atmosphere. The density of the CMCs increased by applying energy field of EM wave, magnetic flux and ultrasonic wave to the reaction atmosphere or substrate as shown later.
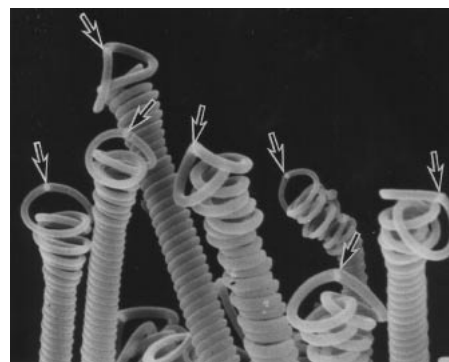

Fig. 1.   Tip of the carbon microcoils grown on the substrate. Reaction time: 5 min. Arrow indicates a catalyst grain.
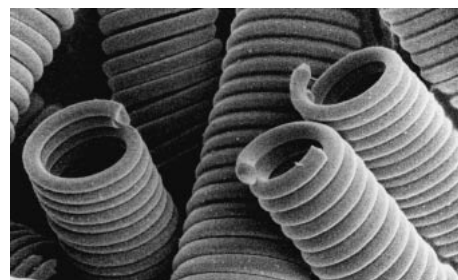

Fig. 2.   Regular carbon microcoils ("circular coils").

3. Results and Discussion

3.1 Morphology and Growth Mechanism.   3.1.1 Deposition Patterns: CMCs grew perpendicularly on the substrate surface with the growth tips pointing in the direction of the source gas inlets, and the thickness of coil layers reached 4–8 mm thick after 2 h reaction time as shown in Fig. 1. The yield of CMCs obtained under optimum reaction conditions was 20–30 mg cm^{-2} substrate.

3.1.2 Morphology: The catalyst grain was always observed on the tip of the CMCs. The micro-coiling morphology was formed by the rotation of the catalyst grain. The coiling (rotating) speed was about one cycle per second around the coil axis. CMCs with various coiling morphology, i.e., regular coils, irregular coils, double coils, single coils, etc., were obtained depending on the reaction conditions. Figure 2 shows representative regular-coiled CMCs. The cross section of the carbon fiber, from which the CMC is formed, is circular or elliptical. We refer to these coils as "circular-coils" hereafter. Another type of regular coil is shown in Fig. 3, in which cross section of the fiber is rectangular or ribbon-like, and we refer to these coils as "flat-coils" hereafter. The coil diameter and coil pitch of these regular carbon coils have a constant value and are on the order of micrometers to several hundred nanometers throughout a piece of carbon coils. These regular coils were obtained under optimum reaction conditions, by which the maximum coil yield was obtained. CMCs with irregular coil diameter and coil pitch, such as shown in Fig. 4, were obtained when the conditions were not optimum, while the coil diameter were in the range of 1–50 μm. Figure 5 shows the irregular coils with a relatively large coil diameter (ca. 40 μm). These coils have very high elasticity. They can stretch up to
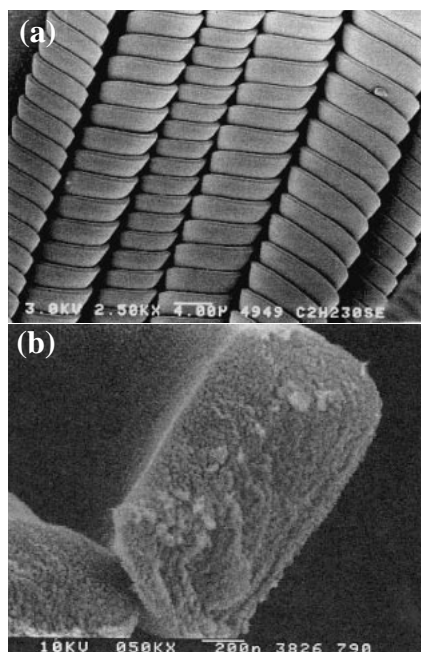
Fig. 3. (a) Regular carbon microcoils ("flat coils"); (b) An enlarged view of the cross section.

Fig. 4. Irregular carbon microcoils.

Fig. 5. Superelastic carbon microcoils with large coil diameter.

10–15 times their original coil length and elastically contract to their original coil length, and thus, they are referred to as super-elastic CMCs. Usually, the coil length was 5–10 mm after 2 h of reaction time. Almost all of the CMCs were double-helix forms in which two fibers entwined with each other such as the double helix of a DNA as can be seen in Fig. 2. The number of right or clockwise carbon coils and left or anticlockwise coils were about the same. The single-helix CMCs with
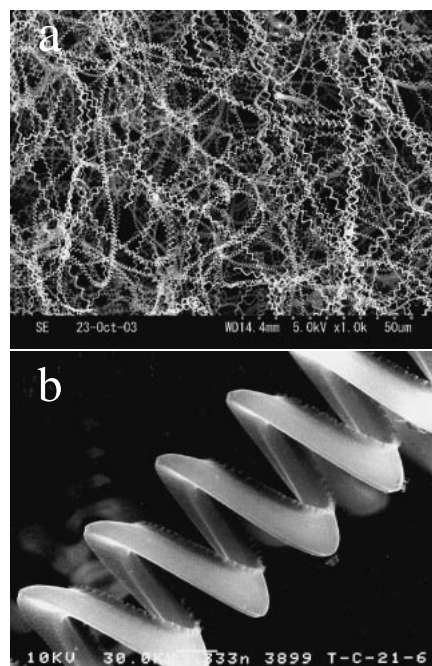

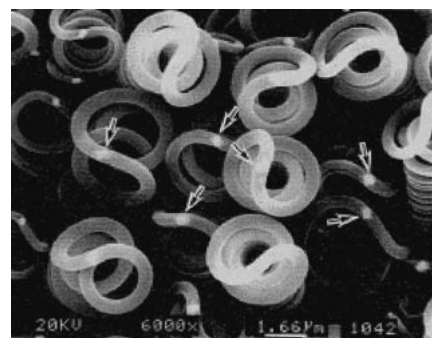Fig. 6. (a) Single-helix CMCs and (b) an enlarged view.

Fig. 7. Tip of the carbon microcoils of initial growth stage. Reaction time: 30 s. Arrow indicates a catalyst grain.

a high purity were also obtained by the selection of the catalysts and controlling of the reaction conditions.[14,15] Figure 6 shows representative single-helix CMCs. Their coil diameter was similar to that of the double-helix CMCs while coil pitch was as large as 1–5 μm.

The carbon wires, from which the CMCs were formed, were generally evenly ruptured, and fine carbon grains were fully filled in the cross section. In addition fine holes, which are found in nanotubes, were not present in the central part of the cross-section of the fibers, while a central tube was observed through the coil axis for the regularly coiled CMCs.

3.1.3 Growth Mechanism: It is very interesting to know why such a peculiar coiling morphology is formed and what is the growth mechanism for the CMCs. Figures 7 and 8 show the tip part of early stage of carbon coils grown over Ni powder catalyst. A bright section (arrow) was always observed on the tip of the CMCs and formed a loop-like shape. XRD analysis and electron diffraction patterns of the catalyst grain showed that the catalyst grain was not a pure Ni metal crystal but a Ni_3C single crystal. In other words, two fibers grew from

Fig. 8. Tip of the carbon microcoils. Arrow indicates a catalyst grain.

Table 1. Effect of Ni Crystals (Single- and Poly-Crystals) on the Deposition of Total Carbon and Coil Yield

	Crystal plane			Polycrystal Ni plate
	Ni(100)	Ni(111)	Ni(110)	
Deposition rate of total carbon /mg cm^{-2}	32	23	19	23
Coil yield /mol %	10.2	6.7	3.2	4.8

the Ni_3C grain (rhombohedral) which was the real catalyst grain. EDX analysis on the Ni_3C catalyst grain showed the presence of a small amount of sulfur and oxygen on the surface, while crystal phases of NiS_x, NiO_x, etc. were not identified. We considered that, using a Ni catalyst, Ni–C–S–O quartenary amorphous thin films are present on the Ni_3C single crystal grain.

A Ni single crystal plate with a (100), (111), (110) plane was used both as a catalyst and the substrate. The effect of the respective crystal face on the coil yield is shown in Table 1. It was observed that different crystal faces grew carbon coils in different coil yield, and the yield order was (100) > (111) > (110). That is, there is large anisotropy of the catalytic effect of the respective crystal faces of the catalyst grain on the coil growth. From these results, we proposed the 3D-growth model of the carbon coils based on the catalytic anisotropy of the crystal faces of the catalyst grain.[16–19] Figure 9 shows the growth model of the circular carbon coils. In this model, the order of the catalytic activity for the carbon deposition among the three crystal faces is A > B > C. Basically, a carbon fiber is composed of fine carbon grains deposited from the three crystal faces of A, B, and C, and curls in such a way that the carbon grains deposited on crystal faces A and B are on the outer side of the coil, while the grains deposited on crystal face C are inside. The coil diameter may be determined by the anisotropy of the carbon deposition between the crystal faces of A and C, and/or B and C, while the coil pitch is determined by that of A and B. We have not yet identified the respective crystal faces; however, faces A, B, and C are most likely (100), (111), and (110), respectively.

3.2 Preparation of Micro-Coils/Micro-Tubes of Metal Carbides and Nitrides.   The carbon coils can be easily metallized and/or nitrided by vapor-phase process and to form micro-coils of metal carbides and/or nitrides with full preserva-
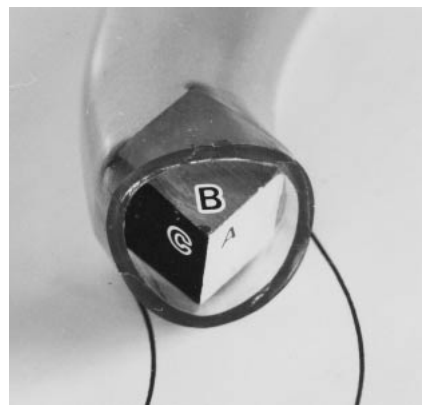

Fig. 9. Schematic image of the growth mechanism for the circular carbon microcoils.
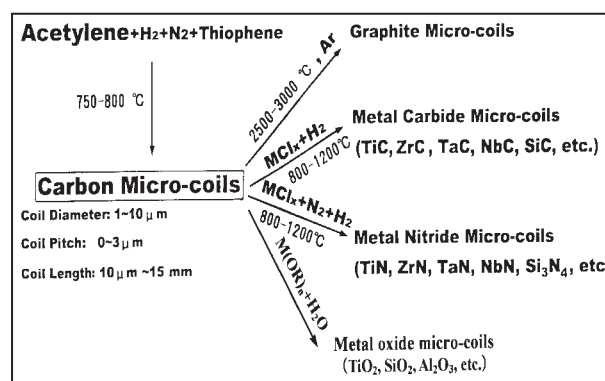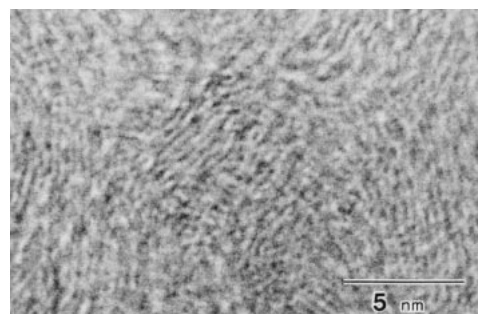

Fig. 10. Modification routes of the carbon.

Fig. 11. TEM image of the CMCs.

tion of the coiling morphology of the CMCs. Using regular-coiled CMCs without coil gap, micro-tubes of MC_x/C(carbon coil)/MC_x–MC_x (MC_x: metal carbide) or MN_x/C/MN_x–MN_x (MN_x: metal nitride) were obtained. These modification processes are shown in Fig. 10.

3.3 Properties.   3.3.1 Composition and Microstructure: The carbon coils were composed of 97.2–98.2 wt % C, 0.6–1.0 wt % O, 1.0–1.4 wt % H, 0.08–0.09 wt % S, and 0.25 wt % Ni. XRD, Raman spectra, selected area electron diffraction patterns, and TEM image (Fig. 11) show that the CMCs are in an amorphous state while very fine films of the outermost surface are in a graphite state.

3.3.2 Density, Specific Surface Area, and Pore Diameter: Table 2 shows the density, specific surface area, and average

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Table 2. Density, Specific Surface Area, and Pore Diameter of the CMCs

External EM field (AC, 60 Hz)	Bias voltage /V	Density /g cm^{-3}	Specific surface area /m^2 g^{-1}	Pore diameter /nm
With	without	1.7234	100–140	3–4
	DC 650	1.8398		
	AC 1300	1.8018		
Without	without	1.7431	70–130	3–4
	DC 650	1.7978		
	AC 1300	1.7901		
Ref. (carbon filters)	nanotube		240	—
	VGCF	2.0817	20–30	
	PAN	1.7402	2–10	
	Pitch	2.0154	1–10	

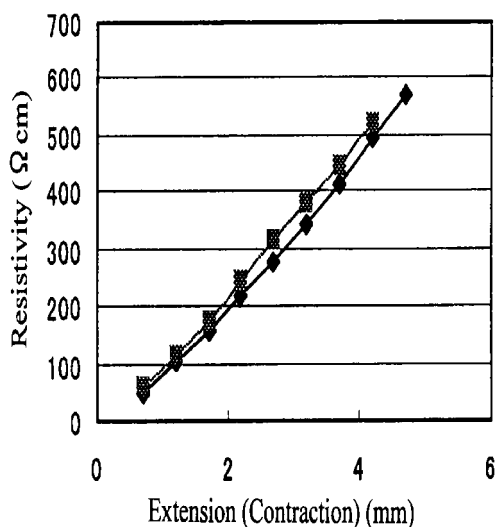Fig. 13. Reflection loss of the CMC (1 wt %)/polyurethane foam sheets. Total thickness: 26 mm.

Fig. 12. The relationship between the extension/contraction of bulk CMCs and electrical resistivity.

pore diameter of the CMCs. Using a conventional electric heater to heat the reaction tube, that is, with EM field, and without bias voltage, the density, specific surface area, and average pore diameter of the CMCs were 1.7234 g cm^{-3}, 100–1400 m^2 g^{-1}, and 2–4 nm, respectively.

3.3.3 Electric Properties:[20] The bulk (powder) electrical resistivity of the CMCs decreased with increasing bulk density and was 1–10 Ω cm for 0.3 g cm^{-3} and 0.1–0.2 Ω cm for 0.6 g cm^{-3}. The electrical resistivity of bulk CMCs increased with increasing extension ratio as shown in Fig. 12, probably caused by an increase in inner stress during stretching. Electrical parameters other than resistivity, such as L (inductance), C (capacitance), X (impedance), θ (phase angle), etc., also changed during stretching and contraction. The resisitivity of the bulk CMCs decreased sharply when the surface was coated with carbon, TiC, TiN, ZrC, NbC, or TaC, but not by graphitizing at high-temperature heat treatment. The electrical conductivity of the CMCs along the helix axis was 30–50 S cm^{-1}. This conductivity is rather small in comparison to multi-wall carbon nanotubes.

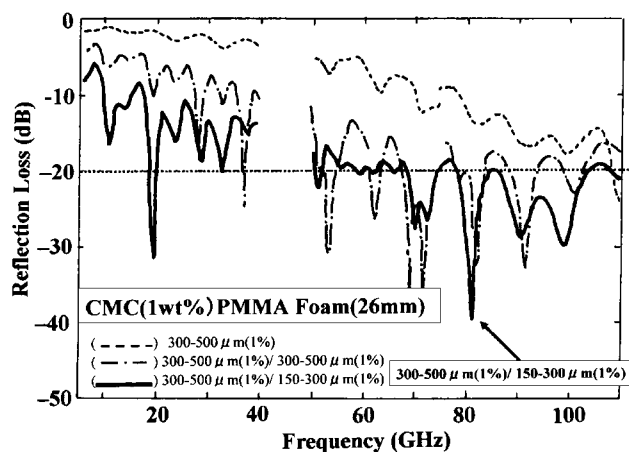**3.3.4 Electromagnetic Properties:**[21–24] It is considered

that the CMCs are promising candidates for use as EM absorbers, especially in the gigahertz (GHz) range, because of its micro-coiling morphology. In other word, the micro-coiling morphology is the most effective and ideal morphology for the generation of an inductive current by Faraday's Law resulting in absorption of EM wave. Actually, CMCs can absorb EM wave in the gigahertz (GHz) region without reflection. Figure 13 shows the reflection loss of PMMA foams with a total thickness of 26 mm in which 1 wt % CMCs was embedded and uniformly dispersed. It can be seen that the reflection loss above −20 dB, which corresponds to 99% absorption, was obtained for double layer of CMCs (300–500 μm)/PMMA// CMCs (150–300 μm)/PMMA over a wide frequency range (50–110 GHz).

3.3.5 Mechanical Properties:[8,12,25–27] Circular CMCs with a large coil diameter could be expanded up to 4.5–15 times original coil length, while flat CMCs could be expanded elastically up to only 1.5 times their original length. The CMCs could be expanded linearly with the increasing in the applied load. The rupture strength of the CMCs was 42–114 MPa. The rigidity of the circular and flat CMCs were 22–46 and 22–33 GPa, respectively. The CMCs were stable under mechanical stirring in water and did not ruptured.

3.3.6 Chemical Properties:[28,29] The CMCs became to oxidize at about 450 °C in air, and the weight significantly decreased with increasing temperature burning out at 700 °C. On the other hand, the graphitized CMCs obtained by heat treatment of the as-grown CMCs at 3000 °C for 6 h in CO + CO$_2$ were oxidized at about 700 °C in air. In order to improve the surface chemical activities, Shibagaki and Motojima examined the partial oxidation characteristics of the surface of the CMCs under a low O$_2$ flow rate at 450–820 °C in detail.[28,29]

3.3.7 Thermal Properties:[30] CMCs could be heat-treated up to 3000 °C in CO + CO$_2$ atmosphere to form graphite coils with a full reservation of the coiling morphology of the source as-grown CMCs. The elasticity of the CMCs was reduced by the heat-treatment, and they became brittle. The graphite coils have a "herringbone" structure in which distinct graphite layers ($d = 0.339$ nm) were developed with an inclination of 35–40 deg versus the fiber axis. The "c" value is slightly larger than the reported value for a graphite of 0.6714 nm while the
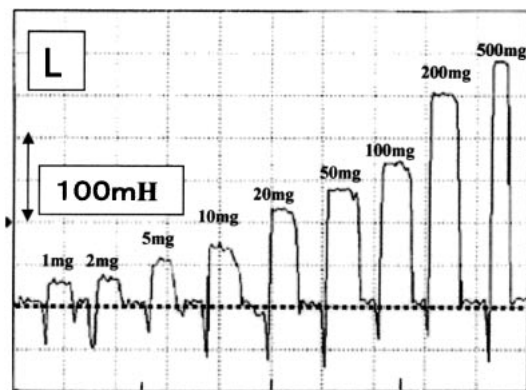
Fig. 14.   The change in L (inductance) parameter of CMC/polysilicone tactile sensor elements under the application of loads. Addition amount of CMC in polysilicone: 1 wt %, thickness of elements: 0.1 mm, separation of electrode: 2.5 mm.

Fig. 15.   Change in L (inductance) parameter of CMC/polysilicone tactile sensor elements without coming a hand directly into contact with it.

"*a*" value is the same as that of graphite ($a = 0.2463$ nm). This may be caused by the presence of disorder in the graphite layers and/or the smaller submicron width graphite layers in the graphite coils.

3.3.8 Tactile Sensing Properties:[27,31]   The CMCs have a high elasticity, and many electrical parameters change upon extension and contraction. The CMCs were embedded into elastic polymers, such as polysilicone, polyurethane, elastic epoxy resin, etc., to form CMC sensor elements, and the change in electrical parameters of the elements under the application of static loads were examined, in which an AC (200 kHz) voltage of 0.5 V was applied to the elements via electrodes. Figure 14 shows the change in the L (inductance) parameter of the CMC (1 wt %)/polysilicone sensor elements with a thickness of 100 μm under the application of small load below 500 mgf. An apparent signal change in the L parameter occurred under a very small load (1 mgf) indicated that the tactile sensor have a very high tactile sensitivity, which is comparable to that of human skin. We also found that the CMC sensor elements can also detect substances without coming directly into contact with it. Figure 15 shows the change in the L parameter without coming a hand directly into contact with it. An apparent signal change is observed at 20 mm separation and very large signal at 1 cm. These properties can be used to prepare highly sensitive tactile and nearness sensors.

3.3.9 Bioactivation: Ogawa has found that CMCs have activation effect for breeding skin cells or collagens; however, the reason is not yet known.[32] For example, the number of skin cells in Pam 212 increased by 1.6 times versus the control after the addition of CMCs (1 μg mL^{-1}). Komura found that CMCs effectively generated hydroxyl radial (•OH) in aqueous solution by ultrasound exposure and can be used in sonodynamic therapy of cancer.[33]

3.4 Applications.   CMCs have many interesting and important characteristics, such as 3D-helical/spiral microstructure with coil diameters on the order of micrometers, diverse microstructures from amorphous to graphite, changing electrical parameters under the application of various stimuli, high microwave absorption and microwave heating abilities, breeding and anti-breeding effect on cells, fibrils of organisms, etc.
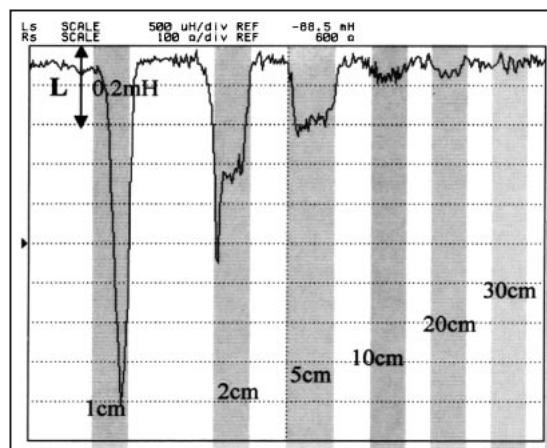
CMCs have been used in commercial applications such as cosmetics, microwave absorbers, etc. In a word, CMCs can be used for electromagnetic wave absorbers, remote heating materials, visualization elements of microwaves, tactile sensor elements, micro antennae, chiral catalysts, bio-activators or bio-deactivators, thermotherapies for cancer, energy converters, etc.

4. Conclusion

CMCs with a coil diameter on the order of micrometers to nanometers were obtained by metal-catalyzed pyrolysis of acetylene, and the preparation conditions, morphology, growth mechanism, and some properties of the carbon coils were examined. CMCs have many interesting and important characteristics, such as 3D-helical/spiral structure with coil diameters on the order of micrometers, diverse microstructures from amorphous to graphite, changing electrical parameters under various stimuli, high microwave absorption and microwave heating abilities, breeding and anti-breeding effect on cells, fibrils of organisms, etc. The micro-coils or micro-tubes of various transition-metal carbides and nitride were obtained by vapor-phase metalizing and/or nitriding of the CMCs with full preservation of the coiling morphology. CMCs are novel functional materials with 3D-helical/spiral forms with a coil diameter on the order of micrometer to nanometer, and have possible uses a electromagnetic absorbers, hydrogen absorbers, tactile and nearness sensor elements, tunable micro/nano devices/sensors, chiral catalysts, thermotherapies for cancers, capacitors, energy converters, etc.

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Award recipient

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