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MIYAGAWA, Hiroaki [JP/US]; 3760 North Way, #23,
Oceanside, California 92056 (US).

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(74) Agent: MALLON, Joseph J.; Knobbe Martens Olson &
Bear LLP, 2040 Main Street, Fourteenth Floor, Irvine, Cal-
ifornia 92614 (US).

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(71) Applicant (*for all designated States except US*): NITTO
DENKO CORPORATION [JP/JP]; 1-1-2 Shimohozumi,
Osaka, Ibaraki 567-8680 (JP).

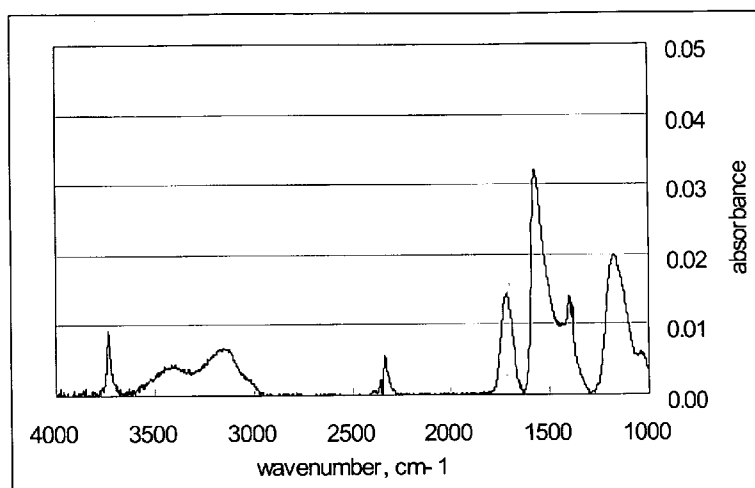
(72) Inventors; and

(75) Inventors/Applicants (*for US only*): JUNI, Noriyuki
[JP/JP]; 1-1-2, Ibaraki-shi, Osaka-fu (JP). MOCHIZUKI,
Amane [US/US]; 11806 Paseo Lucido, #2006, San
Diego, California 92128 (US). NAKAMURA, Toshi-
taka [JP/US]; 3542 Paseo De Los America, Oceanside,
California 92054 (US). GU, Bin [CN/US]; 147 Canyon
Drive, Apt. 225, Oceanside, California 92054 (US).

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(54) Title: METHOD FOR PRODUCING CARBON NANOTUBES, METHOD FOR PRODUCING LIQUID DISPERSION
THEREOF AND OPTICAL PRODUCT



(57) Abstract: A method for producing dispersible carbon nanotubes of the invention comprises subjecting single-walled carbon nanotubes to UV treatment in the presence of ozone such that carboxyl groups are introduced into the single-walled carbon nanotubes and that the single-walled carbon nanotubes are fragmented. A carbon nanotubes obtained by the method has a good dispersibility.

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METHOD FOR PRODUCING CARBON NANOTUBES, METHOD FOR PRODUCING LIQUID DISPERSION THEREOF AND OPTICAL PRODUCT

BACKGROUND OF THE INVENTION

Field of the Invention

[0001] The invention relates to a method for producing dispersible carbon nanotubes. The carbon nanotubes obtained by the production method of the invention have good dispersibility, and highly dispersible carbon nanotubes can be produced according to the invention. The carbon nanotubes of the invention and a liquid dispersion thereof can be used in various applications such as optical products.

Description of the Related Art

[0002] Carbon nanotubes are hollow tubes in which a graphite sheet composed of six-membered carbon rings is rounded into a cylinder with a nanometer-order diameter, and a monolayer cylinder thereof is called a single-walled carbon nanotube, while a multilayer cylinder thereof is called a multi-walled carbon nanotube. Carbon nanotubes have unique excellent properties with respect to high electrical conductivity, mechanical properties, chemical stability, or the like, because of their special structure, and have been investigated for practical use in composite materials, semiconductor devices, electrically-conducting materials, hydrogen absorbing materials, and the like.

[0003] By making use of the properties of carbon nanotubes such as high strength, high modulus, and high electrical conductivity, for example, an attempt has been made in which carbon nanotubes are incorporated as a filler into a polymer material to improve the mechanical properties or electrical conductivity of the polymer material. In order to achieve these, it is necessary to highly disperse carbon nanotubes in a polymer material. However, there is a problem in which it is difficult to disperse carbon nanotubes in a stable manner, due to the van der Waals interaction acting between the carbon nanotubes. When an attempt is made to prepare a liquid dispersion of carbon nanotubes in a solvent, the carbon nanotubes aggregate, and thus it is difficult to obtain a liquid dispersion with good dispersion properties.

[0004] Methods for producing carbon nanotubes with good dispersibility in various polymers or solvents include methods of modifying the surface of carbon nanotubes with hydrophilic groups, such as a method of oxidizing carbon nanotubes with a strong acid. A known method includes allowing carbon nanotubes to react with a strong acid such as nitric acid and sulfuric acid for a sufficient time period to oxidize the surface of the carbon nanotubes and to introduce appropriate hydrophilic groups (see Japanese Patent Application National Publication (Laid-Open) No. 11-502494). There is another method that includes subjecting carbon nanotubes to ultrasonication, while immersing them in a liquid mixture of sulfuric acid and nitric acid, so that the carbon nanotubes are opened and that carboxyl groups are introduced into the open end (see Smalley et al., Science, Vol. 280, pp. 1253-1256 (1988)). However, these methods using strong acids are very dangerous and include time-consuming treatment.

[0005] There is proposed a method that includes applying far ultraviolet light with a wavelength of 1 to 190 nm to carbon nanotubes to make them hydrophilic (see Japanese Patent Application Laid-Open (JP-A) No. 2005-272184). In this method, far ultraviolet light with a wavelength of 172 nm is specifically applied to multi-walled carbon nanotubes in the air so that hydrophilic properties are imparted. However, the hydrophilic carbon nanotubes obtained by this method do not have sufficient dispersibility in solvents or the like, and aggregates are produced due to low dispersibility in a liquid dispersion obtained from the hydrophilic carbon nanotubes. Therefore, it is difficult to use the hydrophilic carbon nanotubes for optical products that require high transparency, because their dispersibility is low.

[0006] There is also proposed a method that includes subjecting multi-walled carbon nanotubes to UV ozone treatment and to triethylenetetramine treatment (see Carbon 44 (2006) 768-777). This method also imparts dispersibility to the multi-walled carbon nanotubes. Since the multi-walled carbon nanotubes treated by this method are for use in forming polymer composites for reinforcement materials and thus have insufficient dispersibility, it is difficult to use such carbon nanotubes for optical products that require high transparency.

SUMMARY OF THE INVENTION

[0007] It is an object of the invention to provide carbon nanotubes with good dispersibility and a method for producing the same.

[0008] It is another object of the invention to provide a liquid dispersion of carbon nanotubes and a method for producing the same.

[0009] It is yet another object of the invention to provide an optical product including the dispersible carbon nanotubes.

[0010] As a result of active investigations for solving the problems, the inventors have found the method described below and made the present invention.

[0011] The present invention related to a method for producing dispersible carbon nanotubes, comprising

subjecting single-walled carbon nanotubes to UV treatment in the presence of ozone such that carboxyl groups are introduced into the single-walled carbon nanotubes and that the single-walled carbon nanotubes are fragmented.

[0012] In the method for producing dispersible carbon nanotubes, the UV treatment in the presence of ozone can perform to apply ultraviolet light having at least two different wavelengths including a wavelength of 240nm or less and a wavelength of 200 to 320 nm in the presence of oxygen so that the UV treatment is performed while the oxygen is converted into ozone.

[0013] In the method for producing dispersible carbon nanotubes, the UV treatment preferably uses an integrated quantity of light of 27 to 324 J/cm².

[0014] In the method for producing dispersible carbon nanotubes, the UV treatment is preferably performed for a time period of 10 minutes to 120 minutes.

[0015] The present invention also related to a dispersible carbon nanotubes produced by the above method.

[0016] The present invention also related to a method for producing a liquid dispersion of carbon nanotubes, comprising:

subjecting single-walled carbon nanotubes to UV treatment in the presence of ozone such that carboxyl groups are introduced into the single-walled carbon nanotubes and that the single-walled carbon nanotubes are fragmented, in order to produce dispersible carbon

nanotubes, which is according to the above method for producing dispersible carbon nanotubes; and

then dispersing the dispersible carbon nanotubes in a solvent.

[0017] The present invention also related to a liquid dispersion of carbon nanotubes produced by the above method.

[0018] The present invention also related to an optical product, comprising the above dispersible carbon nanotubes.

[0019] As the above optical product, the products that the dispersible carbon nanotubes form a coating in a surface of the optical product can be used. The optical product that the optical product with the coating of the dispersible carbon nanotubes product preferably has a surface with a surface resistivity of $1.0 \times 10^{10} \Omega/\text{square}$ or less and has an absorptance of 3.0% or less.

[0020] According to the production method of the invention as stated above, single-walled carbon nanotubes are subjected to UV treatment in the presence of ozone so that carboxyl groups are introduced into the single-walled carbon nanotubes to make the single-walled carbon nanotubes hydrophilic and to improve the dispersibility.

[0021] According to the invention, besides the introduction of the carboxyl groups into the single-walled carbon nanotubes, the UV treatment is performed until at least part of the carbon-carbon bonds in the peripheral direction is broken so that the single-walled carbon nanotubes are fragmented. The dispersibility of the single-walled carbon nanotubes is improved by the introduction of the carboxyl groups and further improved by the fragmentation. Conventionally, single-walled carbon nanotubes with no chemical modification have a very strong cohesive force due to the van der Waals force acting between the single-walled carbon nanotubes and thus are hardly dispersed only by adding them to a solvent even if hydrophilicity is imparted by means of carboxyl groups. According to the invention, not only hydrophilicity is imparted by introducing carboxyl groups into the single-walled carbon nanotubes, but also the van der Waals force is reduced by fragmenting the single-walled carbon nanotubes, so that the dispersibility is remarkably increased by these effects.

[0022] The resulting single-walled carbon nanotubes have good dispersibility and thus can form a liquid dispersion with no aggregate when dispersed in a solvent. While

single-walled carbon nanotubes can be fragmented by UV treatment, multi-walled carbon nanotubes have a multilayer wall and thus are hardly fragmented even though the carbon-carbon bonds in the outer layer can be broken by UV treatment.

[0023] The dispersible carbon nanotubes of the invention and the carbon nanotubes dispersion of the invention can be used in various applications and, for example, specifically used for optical products to form antistatic optical products. The resulting antistatic optical products have high transparency and can satisfy required absorptance and surface resistivity.

BRIEF DESCRIPTION OF THE DRAWINGS

[0024] Fig. 1A is a scanning electron micrograph of carbon nanotubes in a coating of an optical product obtained in Example 1;

[0025] Fig. 1B is a photograph showing the dispersion properties of a liquid dispersion of carbon nanotubes obtained in Example 1 on an optical component;

[0026] Fig. 2A is a scanning electron micrograph of carbon nanotubes in a coating of an optical product obtained in Comparative Example 1;

[0027] Fig. 2B is a photograph showing the dispersion properties of a liquid dispersion of carbon nanotubes obtained in Comparative Example 1 on an optical component;

[0028] Fig. 3 is a scanning electron micrograph of carbon nanotubes in a coating of an optical product obtained in Comparative Example 2;

[0029] Fig. 4 is a scanning electron micrograph of carbon nanotubes in a coating of an optical product obtained in Comparative Example 5; and

[0030] Fig. 5 is an IR chart of carbon nanotubes obtained in Example 1.

DETAILED DESCRIPTION OF THE INVENTION

[0031] According to the invention, single-walled carbon nanotubes are subjected to UV treatment in the presence of ozone so that carboxyl groups are introduced into the single-walled carbon nanotubes and that the single-walled carbon nanotubes are fragmented, when dispersible carbon nanotubes are produced.

[0032] In the production method of the invention, single-walled carbon nanotubes are used as a starting material. For example, the single-walled carbon nanotubes to be used preferably have a diameter of 0.4 to 5 nm, a length of 0.5 to 30 μm and an aspect ratio of 100 to 10000. The diameter of the single-walled carbon nanotubes is preferably 3 nm or less, more preferably 2 nm or less, in terms of cleavability. The aspect ratio is preferably from 200 to 5000, more preferably from 500 to 2000, in terms of the light absorptance and dispersibility of films after film formation. The aspect ratio of the produced single-walled carbon nanotubes is smaller than that of the starting material due to the fragmentation, and the ratio between them is preferably 1/2 or less, more preferably 1/10 or less.

[0033] The method of the invention may use any single-walled carbon nanotubes produced by various production methods without particular limitations. For example, single-walled carbon nanotubes produced by CVD methods, arc discharge methods, laser ablation methods, HiPco methods, or the like may be used.

[0034] In the UV treatment in the presence of ozone, the ozone may exist only in the vicinity of the single-walled carbon nanotubes. For example, the UV treatment in the presence of ozone includes applying ultraviolet light having at least two different wavelengths including a wavelength of 240nm or less and a wavelength of 200 to 320 nm in the presence of oxygen so that UV treatment is performed while the oxygen is converted into ozone. A wavelength of 240nm or less is the lower limit of the ultraviolet region.

[0035] Ultraviolet light with a wavelength of 242 nm or less is absorbed by the oxygen so that ozone is produced. In particular, the ultraviolet light preferably has a wavelength of 184.9 nm. On the other hand, ultraviolet light with a wavelength of 200 to 320 nm decomposes ozone. In particular, such ultraviolet light preferably has a wavelength of 253.7 nm. The combination of the two wavelength ranges of ultraviolet light allows continuous production and decomposition of ozone. Atomic oxygen generated upon the production and decomposition of ozone has a strong oxidizing power. The single-walled carbon nanotubes are fragmented by breaking carbon-carbon bonds in the surface when irradiated with the ultraviolet light, and the single-walled carbon nanotubes are also oxidized with the generated atomic oxygen so that carboxyl groups are introduced into them. The reaction in the presence of oxygen may be performed in the air or while oxygen is supplied.

[0036] A lamp capable of emitting ultraviolet light with a wavelength of 240nm or less and ultraviolet light with a wavelength of 200 to 320 nm may be used for the UV ozone treatment. Alternatively, lamps emitting these ultraviolet rays, respectively, may be used in combination. A lamp or lamps that efficiently emit ultraviolet light with a wavelength of 184.9 nm and ultraviolet light with a wavelength of 253.7 nm are preferred. The type of the lamp to be used may be a low-pressure mercury lamp, a medium-pressure mercury lamp, a high-pressure mercury lamp, or the like. The UV ozone treatment is characterized in that a strong oxidative effect is utilized in the process of producing and decomposing ozone. For example, a low-pressure mercury lamp for use in a UV ozone apparatus emits ultraviolet rays mainly with wavelengths of 184.9 nm and 253.7 nm. When oxygen in the air is irradiated with ultraviolet light at 184.9 nm, it absorbs the ultraviolet light to produce ozone. When the ozone is irradiated with ultraviolet light at 253.7 nm, it absorbs the ultraviolet light and is decomposed. In the process of producing and decomposing ozone, atomic oxygen is generated, which has a strong oxidizing power. Besides the above, a Xe excimer lamp emitting light with a wavelength of 172 nm may also be used for the UV ozone treatment. The excimer lamp (only with a wavelength of 172 nm) allows the production of ozone with ultraviolet light but does not allow the decomposition of ozone. The excimer lamp seems to allow direct production of atomic oxygen. However, active oxygen species generated with the excimer lamp can cause an oxidation reaction only in the immediate vicinity of the lamp surface, and the light with a wavelength of around 172 nm is strongly absorbed by oxygen so that the irradiation distance should be short in the air. Thus, in terms of providing stable oxidizability, it is advantageous to use a low-pressure mercury lamp or lamps capable of emitting ultraviolet light with a wavelength of 240nm or less and ultraviolet light with a wavelength of 200 to 320 nm (specifically emitting ultraviolet light with wavelengths of 184.9 nm and 253.7 nm).

[0037] In the UV treatment in the presence of ozone according to the invention, the ozone may only exist when UV is applied, and thus the UV treatment may be performed while ozone is supplied from an ozone generator or the like. In this case, the wavelength of the ultraviolet light may be selected such that at least ozone can be decomposed, carbon-carbon bonds can be broken, and the single-walled carbon nanotubes can be fragmented.

[0038] The single-walled carbon nanotubes may be kept stationary when subjected to the UV treatment. Alternatively, the single-walled carbon nanotubes may be shaken or rotated such that the entire surface of the single-walled carbon nanotubes can be treated, when subjected to the UV treatment.

[0039] The integrated quantity of light in the UV treatment is preferably from 27 to 324 J/cm². With an integrated quantity of light in the above range, carboxyl groups can be introduced into the single-walled carbon nanotubes, and the single-walled carbon nanotubes can be fragmented, so that the dispersibility can be improved. An integrated quantity of light less than 27 J/cm² is so small that the single-walled carbon nanotubes can be insufficiently fragmented. An integrated quantity of light more than 324 J/cm² is not preferred in view of treatment time. The integrated quantity of light in the UV treatment is more preferably from 80 to 240 J/cm², still more preferably from 120 to 200 J/cm².

[0040] The time period of the UV ozone treatment is preferably from 10 to 120 minutes. With a UV treatment time in the above range, carboxyl groups can be introduced into the single-walled carbon nanotubes, and the single-walled carbon nanotubes can be fragmented, so that the dispersibility can be improved. The UV treatment for a time period of less than 10 minutes can be insufficient so that the single-walled carbon nanotubes can be insufficiently fragmented. A UV treatment time of more than 120 minutes is too long and not preferred because such a time period can reduce the productivity. The UV treatment time is more preferably from 30 to 100 minutes, still more preferably from 50 to 80 minutes.

[0041] By the production method described above, dispersible carbon nanotubes are yielded which comprise fragmented single-walled carbon nanotubes having introduced carboxyl groups. The dispersible carbon nanotubes have good dispersibility and can form a liquid dispersion of carbon nanotubes with good dispersion properties when dispersed in a solvent.

[0042] Examples of the solvent for use in the liquid dispersion include dimethylformamide, isopropyl alcohol, water, methyl isobutyl ketone, methyl ethyl ketone, cyclopentanone, ethyl acetate, and butyl acetate. While the concentration of the carbon nanotubes in the dispersion is not particularly limited and appropriately determined depending on applications of the dispersion, it is generally from about 0.01 to about 0.5% by weight, more preferably from 0.03 to 0.2% by weight.

[0043] For example, the dispersible carbon nanotubes of the invention and the liquid dispersion thereof may be used for optical products. When the dispersible carbon nanotubes are used, the surface of an optical component may be coated with the liquid dispersion thereof. Additionally, the dispersible carbon nanotubes of the invention or the liquid dispersion thereof may be mixed with a polymer material or a solution thereof to be incorporated into the polymer material.

[0044] The surface of an optical product having a coating of the dispersible carbon nanotubes can satisfy a surface resistivity of 1.0×10^{10} Ω /square or less and an absorptance of 3.0% or less and thus has high transparency and antistatic properties. The surface resistivity is more preferably 1.0×10^9 Ω /square or less, and the absorptance is more preferably 2.0% or less.

[0045] Examples of the optical product include a display surface for displays, a surface coating material for the displays, a front panel for projection televisions, a coating material for gauges, and an optical information recording medium such as CD and DVD.

EXAMPLES

[0046] Examples of the invention and Comparative Examples are described below. It will be understood that these examples are not intended to limit the scope of the invention. Concerning the resulting carbon nanotubes, the measurement of the COOH content (%) and the evaluation of the dispersibility are performed by the methods below.

Measurement of COOH Content

[0047] The COOH content of the resulting carbon nanotubes was calculated using neutralization titration and according to the formula below. A pH meter was used in the measurement.

$$\text{COOH (\%)} = V_{\text{NaOH}} \times C_{\text{NaOH}} \times 45 \times 100 / M_{\text{CNT}}$$

V_{NaOH} : Volume of NaOH at equivalent point;

C_{NaOH} : Concentration of NaOH solution;

45: Formula weight of COOH;

M_{CNT} : Weight of carbon nanotubes

Dispersibility of Carbon Nanotubes

○: No aggregate was observed in the resulting dispersion.

△: A few aggregates were observed in the resulting dispersion.

×: Aggregates were observed in the resulting dispersion.

Example 1UV Ozone Treatment

[0048] Single-walled carbon nanotubes (1 to 2 nm in diameter and 5 to 30 μm in length, manufactured by Cheap Tubes, Inc.) were subjected to UV treatment in the air for 60 minutes using a UV ozone treatment system (applying ultraviolet light with wavelengths of 184.9 nm and 253.7 nm, manufactured by SAMCO, Inc.). The treatment was performed while ozone was generated by the UV irradiation. The integrated quantity of light was 162 J/cm².

Preparation of Liquid Dispersion

[0049] The single-walled carbon nanotubes resulting from the treatment were dispersed at a concentration of 0.05% by weight in a dimethylformamide (DMF) solvent and subjected to dispersion treatment for 30 minutes in an ultrasonic dispersion machine (Fisher Model 100 Sonic Dismembrator) to form a liquid dispersion. The resulting liquid dispersion was a carbon nanotubes dispersion with very good dispersion properties. The resulting liquid dispersion was immediately observed with an optical microscope (at a magnification of 100 $\times$), and no aggregate of carbon nanotubes was observed.

Optical Product Application

[0050] The resulting liquid dispersion was applied onto a glass substrate by spin coating such that the coating would have a thickness of 10 nm or less after drying, and then the coating was heated and dried at 100°C for 2 minutes so that an optical product with a carbon nanotubes coating was obtained.

[0051] The surface of the optical product was observed with a scanning electron microscope (HITACHI S-4800, at a magnification of 50000 $\times$), and it was observed that the structure of the carbon nanotubes were fragmented. The result is shown in Fig. 1A. The

dispersion state of the resulting carbon nanotubes dispersion is shown in the photograph of Fig. 1B. The resulting carbon nanotubes were analyzed with an FTIR system (Magna 750/F-Thunderdome, Thermo Nicolet), and a peak derived from carboxyl groups was observed at about 1720 cm^{-1} . The FTIR chart is shown in Fig. 5.

[0052] The resulting optical product was evaluated as described below.

Absorptance

[0053] The total light transmissions of the glass substrate and the optical product with the carbon nanotubes coating were measured with a haze meter (HM-150, manufactured by Murakami Color Research Laboratory), and the absorptance was determined from the result. The absorptance is based on the absorption caused by adding the carbon nanotubes and is a difference between the total light transmissions of the resulting optical product and the substrate, wherein a base line is set for an optical component with no carbon nanotubes (the glass substrate). The resulting absorptance was 1.91%.

Surface Resistivity

[0054] The surface resistivity of the optical product with the carbon nanotubes coating was measured with a resistivity meter (Hiresta MCP-HT450, manufactured by Dia Instruments Co., Ltd.). The resulting surface resistivity was $8.8 \times 10^6\ \Omega/\text{square}$.

Example 2

Preparation of Liquid Dispersion

[0055] A liquid dispersion of carbon nanotubes was obtained using the process of Example 1, except that the amount of the added single-walled carbon nanotubes resulting from the UV ozone treatment was set at 0.03% by weight in the preparation of the liquid dispersion according to Example 1. The resulting liquid dispersion was immediately observed with an optical microscope, and no aggregate of carbon nanotubes was observed.

Optical Product Application

[0056] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Example 3Preparation of Liquid Dispersion

[0057] A liquid dispersion of carbon nanotubes was obtained using the process of Example 1, except that the amount of the added single-walled carbon nanotubes resulting from the UV ozone treatment was set at 0.025% by weight in the preparation of the liquid dispersion according to Example 1. The resulting liquid dispersion was immediately observed with an optical microscope, and no aggregate of carbon nanotubes was observed.

Optical Product Application

[0058] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Example 4Preparation of Liquid Dispersion

[0059] A liquid dispersion of carbon nanotubes was obtained in the same manner as Example 1. The resulting liquid dispersion was immediately observed with an optical microscope, and no aggregate of carbon nanotubes was observed.

Optical Product Application

[0060] The carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. Thereafter, a UV-curable acrylic resin (Unidic 17-806, manufactured by Dainippon Ink and Chemicals, Incorporated) was applied thereto by spin coating such that the coating would have a thickness of 5 μm after drying. The coating was heated and dried at 100°C for 2 minutes and then UV-cured so that an optical product with the carbon nanotubes coating was obtained. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Comparative Example 1

Preparation of Liquid Dispersion

[0061] Single-walled carbon nanotubes (1 to 2 nm in diameter and 5 to 30 μm in length, manufactured by Cheap Tubes, Inc.) were ultrasonically dispersed at a concentration of 0.05% by weight in a DMF solvent to form a carbon nanotubes dispersion. The resulting liquid dispersion was immediately observed with an optical microscope, and aggregates of the carbon nanotubes were observed.

Optical Product Application

[0062] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The structure of the surface of the optical product was observed with a scanning electron microscope. The result is shown in Fig. 2A. The state of the resulting carbon nanotubes dispersion is also shown in the photograph of Fig. 2B. It is apparent that the dispersion properties are worse in Fig. 2B than in Fig. 1A. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Comparative Example 2

Preparation of Liquid Dispersion

[0063] Multi-walled carbon nanotubes (8 nm in diameter and 10 to 30 μm in length, manufactured by Cheap Tubes, Inc.) were ultrasonically dispersed at a concentration of 0.05% by weight in a DMF solvent to form a carbon nanotubes dispersion. The resulting liquid dispersion was immediately observed with an optical microscope, and aggregates of the carbon nanotubes were observed.

Optical Product Application

[0064] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The structure of the surface of the optical product was observed with a scanning electron microscope. The result is shown in Fig. 3. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Comparative Example 3

UV Ozone Treatment

[0065] UV ozone treatment was performed using the process of Example 1, except that multi-walled carbon nanotubes (8 nm in diameter and 10 to 30 μm in length, manufactured by Cheap Tubes, Inc.) were used in place of the single-walled carbon nanotubes.

Preparation of Liquid Dispersion

[0066] The multi-walled carbon nanotubes resulting from the treatment were ultrasonically dispersed at a concentration of 0.025% by weight in a DMF solvent to form a liquid dispersion. The resulting liquid dispersion was immediately observed with an optical microscope, and a few aggregates of the carbon nanotubes were observed.

Optical Product Application

[0067] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The structure of the surface of the optical product was observed with a scanning electron microscope. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Comparative Example 4Preparation of Liquid Dispersion

[0068] A liquid dispersion of carbon nanotubes was obtained using the process of Comparative Example 3, except that the amount of the added multi-walled carbon nanotubes resulting from the UV ozone treatment was set at 0.03% by weight in the preparation of the liquid dispersion according to Comparative Example 3. The resulting liquid dispersion was immediately observed with an optical microscope, and a few aggregates of the carbon nanotubes were observed.

Optical Product Application

[0069] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The structure of the surface of the optical product was observed with a scanning electron microscope. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Comparative Example 5Preparation of Liquid Dispersion

[0070] A liquid dispersion of carbon nanotubes was obtained using the process of Comparative Example 3, except that the amount of the added multi-walled carbon nanotubes resulting from the UV ozone treatment was set at 0.05% by weight in the preparation of the liquid dispersion according to Comparative Example 3. The resulting liquid dispersion was immediately observed with an optical microscope, and aggregates of the carbon nanotubes were observed.

Optical Product Application

[0071] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The structure of the surface of the optical product was observed with a scanning electron microscope. The result is shown in Fig. 4. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Comparative Example 6Preparation of Liquid Dispersion

[0072] A liquid dispersion of carbon nanotubes was obtained using the process of Example 1, except that the time period of the UV treatment was alternatively set at 1 minute. The integrated quantity of light was 162 J/cm^2 . The resulting liquid dispersion was immediately observed with an optical microscope, and aggregates of the carbon nanotubes were observed.

Optical Product Application

[0073] The resulting carbon nanotubes dispersion was used to form an optical product with a carbon nanotubes coating in the same manner as Example 1. The structure of the surface of the optical product was observed with a scanning electron microscope; and there was no change in the carbon nanotubes structure from that shown in Fig. 3. The result is shown in Fig. 4. The resulting product was evaluated in the same manner as Example 1 (for absorptance and surface resistivity). The results are shown in Table 1.

Table I

	Structure of Carbon Nanotubes	Treatment Conditions			COOH Content (%)	Dispersibility of Carbon Nanotubes			
		Atmosphere	Time Period of UV Treatment	Integrated Quantity of Light (J/cm²)			Amount of Added Carbon Nanotubes	Absorptance (%)	Surface Resistivity (Ω/square)
Example 1	single-walled	ozone	60 min	162	3.5	○	0.05	1.91	8.8×10E6
Example 2	single-walled	ozone	60 min	162	3.5	○	0.03	1.41	3.1×10E7
Example 3	single-walled	ozone	60 min	162	3.5	○	0.025	0.87	1.1×10E8
Example 4	single-walled	ozone	60 min	162	3.5	○	0.05	1.54	9.2×10E7
Comparative Example 1	single-walled	none	none	none	0	×	0.05	6.09	3.0×10E12
Comparative Example 2	multi-walled	none	none	none	0	×	0.05	6.41	1.0×10E12
Comparative Example 3	multi-walled	ozone	60 min	162	1.5	△	0.025	0.9	1.1×10E10
Comparative Example 4	multi-walled	ozone	60 min	162	1.5	△	0.03	1.7	2.0×10E10
Comparative Example 5	multi-walled	ozone	60 min	162	1.5	×	0.05	3.8	6.4×10E7
Comparative Example 6	multi-walled	ozone	1 min	2.7	Outside limit of measurement	×	0.05	5.98	3.0×10E12

[0074] In Examples, the dispersibility is good so that the absorptance and surface resistivity of each optical product are both small, because carboxyl groups are introduced into the single-walled carbon nanotubes and the single-walled carbon nanotubes are fragmented. In Comparative Examples, the dispersibility was poor, because the single-walled or multi-walled carbon nanotubes are not fragmented. In Comparative Example 3 or 4, the dispersibility is improved to some extent by the introduction of carboxyl groups, but a few aggregates are observed. In Comparative Example 5, carboxyl groups are also introduced similarly to Comparative Example 3 or 4, but the dispersibility is poor and aggregates are observed, because the conditions of the UV ozone treatment (the amount of the added carbon nanotubes) are the same as those of Example 1. In this regard, it is apparent that the Examples should also be suitable for UV ozone treatment at high concentrations. In Comparative Example 6, the content of COOH was less than the measuring limit, because the time period of the UV ozone treatment was short.

WHAT IS CLAIMED IS:

1. A method for producing dispersible carbon nanotubes, comprising
subjecting single-walled carbon nanotubes to UV treatment in the presence of ozone such that carboxyl groups are introduced into the single-walled carbon nanotubes and that the single-walled carbon nanotubes are fragmented.
2. The method for producing dispersible carbon nanotubes according to Claim 1, wherein the UV treatment in the presence of ozone comprises applying ultraviolet light having at least two different wavelengths including a wavelength of 240nm or less and a wavelength of 200 to 320 nm in the presence of oxygen so that the UV treatment is performed while the oxygen is converted into ozone.
3. The method for producing dispersible carbon nanotubes according to any of Claims 1 to 2, wherein the UV treatment uses an integrated quantity of light of 27 to 324 J/cm².
4. The method for producing dispersible carbon nanotubes according to any of Claims 1 to 3, wherein the UV treatment is performed for a time period of 10 minutes to 120 minutes.
5. A dispersible carbon nanotube produced by the method for producing dispersible carbon nanotubes according to any of Claims 1 to 4.
6. A method for producing a liquid dispersion of carbon nanotubes, comprising:
subjecting single-walled carbon nanotubes to UV treatment in the presence of ozone such that carboxyl groups are introduced into the single-walled carbon nanotubes and that the single-walled carbon nanotubes are fragmented, in order to produce dispersible carbon nanotubes; and
then dispersing the dispersible carbon nanotubes in a solvent.
7. The method for producing a liquid dispersion of carbon nanotubes according to Claim 6, wherein the UV treatment in the presence of ozone comprises applying ultraviolet light having at least two different wavelengths including a wavelength of 240nm or less and a wavelength of 200 to 320 nm in the presence of oxygen so that the UV treatment is performed while the oxygen is converted into ozone.

8. The method for producing a liquid dispersion of carbon nanotubes according to any of Claims 6 to 7, wherein the UV treatment uses an integrated quantity of light of 27 to 324 J/cm².

9. The method for producing a liquid dispersion of carbon nanotubes according to any of Claims 6 to 8, wherein the UV treatment is performed for a time period of 10 minutes to 120 minutes.

10. A liquid dispersion of carbon nanotube produced by the method for producing a liquid dispersion of carbon nanotubes according to any of Claims 6 to 9.

11. An optical product, comprising the dispersible carbon nanotubes according to Claim 5 or according to Claim 10.

12. The optical product of Claim 11, wherein the dispersible carbon nanotubes form a coating in a surface of the optical product.

13. The optical product of Claim 12, wherein the optical product with the coating of the dispersible carbon nanotube product has a surface with a surface resistivity of 1.0×10^{10} Ω /square or less and has an absorptance of 3.0% or less.

FIG.1A

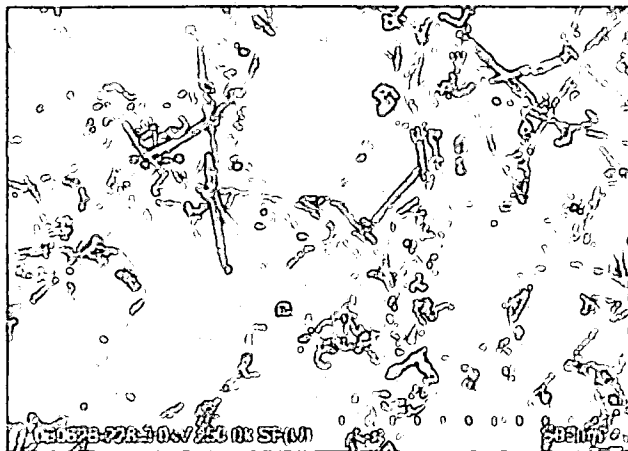


FIG.1B



FIG.2A

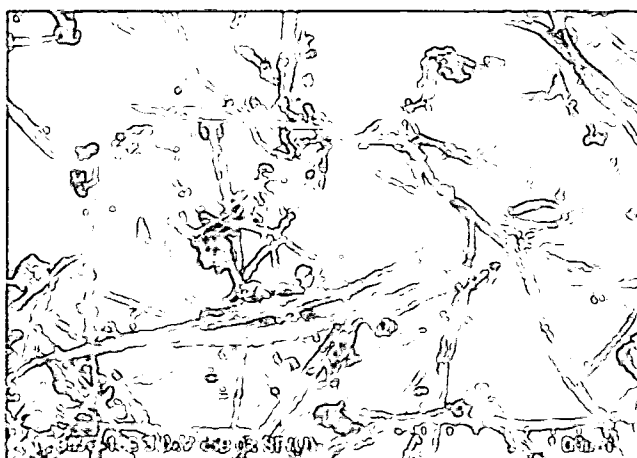


FIG.2B



FIG.3

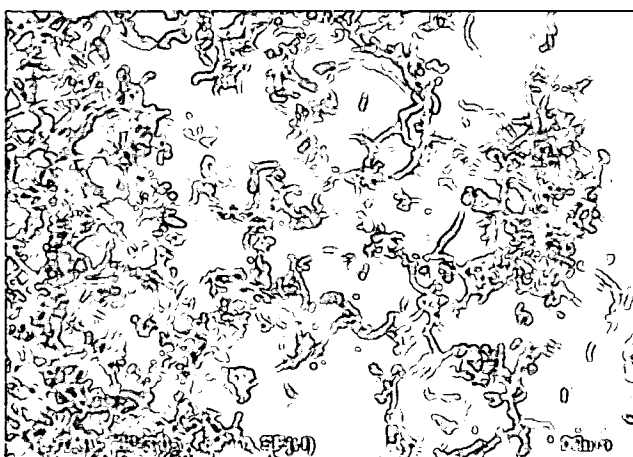
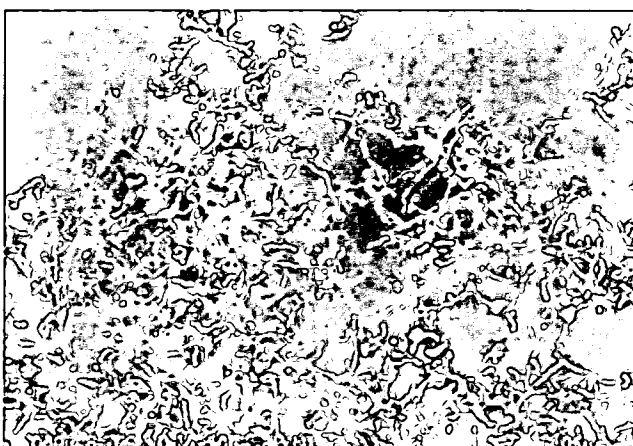


FIG.4



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FIG.5