

DESCRIPTION

METHOD OF PRODUCTION OF NANO-MATERIALS BY VAPOR EXPLOSION,
APPARATUS THEREFOR AND METHOD OF PRODUCTION OF
5 NANO-SOURCE-MATERIALS

TECHNICAL FIELD

The present invention relates to a method for production of nano-materials, by which nano-materials can be produced with
10 facility, in high efficiency and at low cost, and an apparatus therefor as well as a method for production of source materials therefor.

BACKGROUND ART

15 As methods for production of nano-materials such as fullerenes and carbon-nanos, three methods of a laser evaporation method, an arc discharge method and a CVD (Chemical Vapor Deposition) method have been hitherto known.

The laser evaporation method is a production method by which
20 graphite heated to 1200 °C in an electric furnace is evaporated by irradiation of a laser beam of a carbon dioxide laser or an excimer laser and then fullerenes and such are produced on a furnace inner wall. In a case of not mediating catalytic metal, fullerenes and nano-tubes are obtained, whereas in a case of mediating
25 catalytic metal, metallofullerenes and such are obtained.

The arc discharge method is another production method by which an arc discharge is generated between electrodes of graphite and carbon atoms are evaporated from the electrodes. Similarly to the above, depending on existence of catalytic metal, fullerenes
30 and nano-tubes or metallofullerenes are generated and adhere to the furnace inner wall.

The CVD method is still another production method by which fine particles of catalytic metal are mixed in a hydrocarbon gas and introduced into an electric furnace from 600 °C to 1000 °C
35 and then nano-tubes are obtained by assembling carbon atoms.

Information of related arts is disclosed in Separated Volume of Nikkei Science, vol. 138, pages 22-27 (published by Nihon Keizai

Shimbun, Inc. on October 9, 2002).

DISCLOSURE OF INVENTION

Any of the aforementioned production methods merely allows
5 a production in a laboratory level and hence may not be considered
an efficient production method. Even the CVD method, which is
now considered as the most productive method, needs a process for
removing the catalytic metal and hence may not be considered
sufficiently efficient. Furthermore, it puts a tight restriction
10 on the source material and has a problem with quality of the produced
nano-tubes.

On the other hand, an inert gas laser method, by which an
inert gas is filled in a container, a high-voltage and a
high-temperature are generated by employing electrodes and then
15 a material is decomposed so as to be nano-scaled, has been hitherto
considered to be the most practical production method. A method
by employing a YAG laser and an excimer laser method by which a
short wavelength ray is emitted are besides proposed. However,
these methods have a problem that a large apparatus is necessary
20 whereas production is small. Moreover, by these methods, the
source material is evaporated at a high temperature by a laser
irradiation and hence it causes a problem that a fugitive part
thereof is great and an efficiency of capture is low.

Furthermore, a mill is employed for mechanical crushing of
25 carbon, however, there are problems that it takes long hours and
collection of fine powders is difficult.

Specifically, the prior production methods have a problem
of low productivity.

The present invention has been achieved in view of the above
30 problem and is intended for providing a method of production of
nano-materials and an apparatus therefor, by which nano-materials
having high quality can be safely and efficiently produced with
utilizing a simple equipment.

The present inventors are given ideas from various phenomena
35 in the course of achieving the present invention. For example,
when removing deformation of a steel structure, cooling water is
poured thereon. Then it had been observed that the cooling water

did small water vapor explosions on a surface of the steel and mill scales of the steel scatters out with bubbles of the boiling water. It had been also observed that the atmospheric temperature was not so high. In view of these observations, an idea that such
5 water vapor explosions might be employed for obtaining a material as very fine particles had occurred to the present inventors.

With respect to a method for capturing or collecting the nano-material, the present inventors had got an idea in the following way. When taking a bath, the present inventors had
10 observed a state that clouds of steam rose in the bathroom. An idea that very fine water drops composing the clouds of the steam might be employed for capturing or collecting the nano-material had occurred to the present inventors.

More specifically, the present inventors had reached an art
15 in which a water vapor explosion is set off in a tightly enclosed space so as to make powders into fine particles and subsequently the fine particles are efficiently captured by a hot water curtain filling the space.

Next, with respect to a method for setting off the water
20 vapor explosion, the present inventors had got an idea in the following way. In general, any change given to a mixture in temperature or pressure may cause chemical reaction. For example, a waste tire is a mixture of more than 200 kinds of substances. It is observed that such the waste tire is made to be a powder,
25 water is added thereto, they are heated from outside and then the powder of the waste tire undergoes chemical reaction so as to generate water vapor by reaction heat thereof. The present inventors had guessed that nano-materials might be generated by giving rise to such phenomenon in a tightly enclosed space in a
30 relatively moderate atmosphere of a temperature from 100 °C to 200 °C and a pressure of several atoms.

The present inventors had achieved an experiment with using a commercially available autoclave. Powder of a waste tire and some water were pooled in the autoclave and heated from the outside.
35 When the water started boiling and the amount of the water was decreased, the inventors loosened a lid of the autoclave and then an explosion happened therein with a sound of "BONG". After

repeating this procedure several times, the present inventors opened the lid of the autoclave so as to investigate the inner side thereof and discovered a group of the glittering fine particles adhered on the inner wall thereof. The present inventors requested
5 Industrial Research Center of Ehime Prefecture to analyze the group of the fine particles and then identified them to be nano-materials.

The present inventors had further achieved experiments. In an attempt to achieve experiments with an inner lid having a lot of holes installed in the autoclave, the present inventors had
10 discovered that nano-materials were adhered on both sides of the inner lid and hence a yield was increased. The present inventors had further discovered that kinds of the nano-materials varied depending on sites of adhesion, namely, a lower surface of the upper lid, an upper surface of the inner lid, a lower surface of
15 the inner lid and the inner surface of the autoclave.

From the aforementioned discoveries, a law underlying the present invention can be described as follows. A liquid contained in the source material is heated so as to be a saturated state. Then an outer surface of the source material is in a condition
20 of being covered by the hot water curtain and a disturbance is given to the saturated state to break a balance thereof so that a water vapor explosion can be set off. The source material is exposed to the explosion so as to become a fine powder and is dispersed with the vapor. In the dispersing hot water curtain, the fine
25 powder undergoes a chain of explosions so as to become a finer powder and finally becomes carbon clusters having high reactivity. Here the term "carbon cluster" is used as a meaning including an atom, a molecule and a particle, in which plural atoms or molecules gather, of carbon and carbon compound which can react to generate
30 nano-materials. Accompanying with the water vapor explosion, the hot water curtain first diffuses out and next condenses so as to come back to a saturated state. Accordingly, the carbon clusters do not keep diffusing as simple substances but are captured by the hot water curtain and are more definitely captured by cores
35 thereof. The hot water curtain diffuses equally from the view of a start point of the explosion so that carbon clusters having relatively greater weight gather at relatively nearer sites from

the start point and carbon cluster having relatively smaller weight gather at relatively farer cites from the start point. Therefore the nano-materials are accumulated in such a way that the kinds thereof vary depending on distances. Thereby according to the
5 production method of the present invention it is unnecessary to separate the products after production.

According to the first aspect of the present invention, a method of production of a nano-material comprises mixing a substance which is to be a carbon source and a liquid so as to form a source
10 material; heating the source material in a pressure vessel to around a boiling point so as to be in an equilibrium state; giving a disturbance to the equilibrium state so as to set off a water vapor explosion; and making the nano-materials adhere to an inner wall of the pressure vessel. Preferably, the method further comprises
15 injecting liquid to the heated source material, relieving a pressure of the heated source material or releasing the source material in an over-heated state into the vessel through an inflation valve.

According to a second aspect of the present invention, a nano-material production apparatus comprises a pressure vessel;
20 a heating device for heating a source material disposed in the pressure vessel; a capture device disposed in the pressure vessel; a pressure regulation device for regulating a pressure in the pressure vessel; and a control device timely operating the injection device. Preferably, the pressure vessel is an autoclave and the
25 heating device is configured to heat a portion of the autoclave, which receives the source material, or a whole of the autoclave. More preferably, a bottom of the autoclave is configured to receive the source material and the capture device is disposed above the bottom so as to partition the autoclave into an upper half and
30 a lower half. Still preferably, the capture device is a plate having plural through holes. Further preferably, the pressure vessel is cylindrically shaped and horizontally disposed and comprises an open-and-closable lid on at least one end thereof and plural capture members in rows being capable of being taken
35 out and put in the pressure vessel.

According to a third aspect of the present invention, a method of production of a source material for production of the

nano-material comprises dry-distilling a resin at a temperature in a range from 350 to 400 °C; removing an impurity from a residual substance formed by dry-distilling; oxidation-removing an unnecessary substance from the residual substance; and regulating
5 a grain size of the residual substance. Preferably, the method further comprises adding a metal powder.

BRIEF DESCRIPTION OF DRAWINGS

Fig. 1 is a side view of a drum type nano-material production
10 apparatus according to a first embodiment of the present invention;

Fig. 2 is a sectional view of a drum type nano-material production apparatus according to a second embodiment of the present invention;

Fig. 3 is a plan view of an inner lid of the drum type
15 nano-material production apparatus according to the second embodiment of the present invention;

Fig. 4 is a plan view of a cylinder type nano-material production apparatus according to a third embodiment of the present invention;

Fig. 5 is a front view of the cylinder type nano-material production apparatus according to the third embodiment of the present invention;
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Fig. 6 is a schematic drawing showing a former step of a process of a method for production of source material of the
25 nano-material;

Fig. 7 is a schematic drawing showing a later step of the process of the method for production of the source material of the nano-material;

Fig. 8 is an electron microscopic image of the nano-material
30 of an embodiment example of the present invention;

Fig. 9 is an electron microscopic image of the nano-material of an embodiment example of the present invention;

Fig. 10 is an electron microscopic image of the nano-material of an embodiment example of the present invention;

Fig. 11 is an electron microscopic image of the nano-material
35 of an embodiment example of the present invention;

Fig. 12 is an electron microscopic image of the nano-material

of an embodiment example of the present invention;

Fig. 13 is an electron microscopic image of the nano-material of an embodiment example of the present invention; and

Fig. 14 is an electron microscopic image of the nano-material
5 of an embodiment example of the present invention.

BEST MODE FOR CARRYING OUT THE INVENTION

Embodiments of the present invention will be described with reference to the drawings hereinafter.

10 A nano-material production apparatus 1 according to a first embodiment of the present invention is, as shown in Fig. 1, provided with a pressure vessel 2 of a drum type, an outer cylinder 3 surrounding the pressure vessel 2.

The pressure vessel 2 is provided with an open-and-closable
15 lid 2A and they are fixed with each other by means of flanges 4U and 4D thereof and bolts tightened therein. The pressure vessel 2 is configured so that a source material 5 for production of the nano-material can be disposed therein.

The lid 2A is provided with a safety valve 6, a pressure
20 gauge 7 provided with a valve, a liquid injection nozzle 8 which is capable of injecting water into the pressure vessel 2, a temperature sensor 9 and a pressure relief device 10 for pressure relief. A liquid regulation valve 11 is connected to the injection nozzle 8 and a hose 12 is further connected thereto so that water
25 can be introduced from an outside into the pressure vessel 2.

An upper side of the outer cylinder 3 is capable of being closed with a lid 15. A lower portion of the outer cylinder 3 is provided with a heat source introduction port 13 for introduction of heat sources such as vapor or heating gas and, as corresponding
30 thereto, the lid 15 is provided with a heat source exhaust port 14. The lid 15 is further fixed with the outer cylinder 3 by means of flanges and bolts. The pressure vessel 2 is housed as being surrounded by the outer cylinder 3 and the lid 15.

When the lid 2A is opened, the source material 5 can be charged
35 in the pressure vessel 2. When the lid 2A is closed, the pressure vessel 2 can be tightly closed. A side surface of the pressure vessel 2 and a side surface of the outer cylinder 3 may be

appropriately provided with windows for access to the interior thereof. The outer cylinder 3 also has a function of heat-insulation at a time of heating the pressure vessel 2.

In the drum type nano-material production apparatus 1 as
5 such constituted, the source material 5 is charged into the pressure vessel 2. The vapor or the heating gas is blown into the heat source introduction port 13 so as to heat the pressure vessel 2 and the source material 5 up to, for example, 200 °C.

Then a small amount of water is evaporated in the pressure
10 vessel 2 so that the water vapor fills the interior thereof and the surfaces of the source material 5 are in a condition of being covered by the hot water curtain 5C. The internal pressure is regulated in a proper pressure, for example, 0.4 MPa by means of the pressure relief device 10.

15 Next, when a proper amount of water is injected into the pressure vessel 2 through the liquid injection nozzle 8, the water comes into contact with the source material 5 and a bottom of the pressure vessel 2 so that a disturbance is given to a saturated state so that a water vapor explosion is brought off. Thereby,
20 the hot water curtain 5C explosively diffuses upward in the vessel 2 with a sound.

The hot water curtain 5C involves the water vapor and the carbon clusters including super-fine particles formed from the crushed source material 5. In a case where the source material
25 5 includes what may become nuclei, such as fine particles of iron, the carbon clusters are captured by the nuclei to form nano-materials and adheres to the inner surface of the pressure vessel 2. In a case where the source material 5 includes such nuclei, the inner surface of the pressure vessel 2 function as
30 nuclei so as to form the nano-materials.

As the aforementioned description, according to the first embodiment of the present invention, the powdery source material 5 is put in and tightly enclosed in the pressure vessel 2 which is capable of being easily opened, the pressure vessel 2 is heated
35 from the outside, the appropriate amount of the water is injected into the pressure vessel 2 and the water vapor explosion is set off in the pressure vessel 2 so that the powdery source material

5 becomes super-fine particles. In the hot water curtain 5C generated by the water vapor explosion, the source material 5 contained therein further undergoes a secondary explosion in a manner of a chain reaction. The hot water curtain 5C diffuses
5 out to fill the pressure vessel 2 and the water vapor captures the dispersed super-fine particles by means of a condensation function of the water vapor. Thereby, nano-carbons can be formed in an atmosphere having a relatively low temperature about 100 - 200 °C. The water vapor explosion can be repeatedly set off
10 by opening the liquid regulation valve 11 so as to inject the appropriate amount of the water into the pressure vessel. Thereby, productivity can be increased. Since making the source material 5 into the super-fine particles and capturing the super-fine particles by the hot water curtain 5C are simultaneously done,
15 a simple and highly productive production can be put into practice. Moreover, crystallization of a super-carbon can be achieved. Changing quality of the carbon can be avoided and a great amount of high carbon can be produced by means of a low-cost equipment. Nano-carbons produced by the present invention can be widely applied
20 to advanced industrial fields.

The drum-type nano-material production apparatus according to the first embodiment of the present invention can be modified into a second embodiment shown in Figs. 2 and 3.

According to the second embodiment, a flange 4D of a pressure
25 vessel 2 projects inward and an inner lid 16 is disposed thereon. The inner lid 16 is provided with a circular plate 18 having plural holes 17 made of a punching metal and a circular support frame 19 surrounding the circular plate 18. The inner lid 16 is configured to be fixed to the flange 4D. A slit 20 having an appropriate
30 width may be provided between the circular plate 18 and the support frame 19.

Similar to the description of the first embodiment, the source material 5 and water are put in and tightly enclosed in the open-and-closable pressure vessel 2 and the pressure vessel 2 is
35 heated from the outside. After the temperature and the pressure in the pressure vessel 2 reach appropriate values, the internal pressure is suddenly decreased by operation of the pressure relief

device 10. Then the saturated state is disturbed as mentioned above and hence it leads to the water vapor explosion. Thereby the hot water curtain 5C explosively diffuses upward in the vessel 2 with a sound. In the hot water curtain 5C generated by the water vapor explosion, the source material 5 contained therein further undergoes a secondary explosion in a manner of a chain reaction. Thereby, nano-materials can be formed similarly to the aforementioned description. The formed nano-materials adhere to the inner wall of the pressure vessel 2 and the inner lid 16. The formation of the nano-materials can be repeatedly achieved by repeating operation of decrease in the internal pressure in the pressure vessel 2. Depending on kinds of the source materials, various types of the nano-materials such as carbon-nano, material-nano, plastic-nano, rubber-nano and so on can be formed. These can be applied to electric or electrical parts, tablewares and medical treatments.

The inner lid 16 is provided with the slid 20 and the plural holes 17 and the hot water curtain 5C is capable of passing therethrough. More specifically, the nano-materials transported by the hot water curtain 5C may be captured by both surfaces of the inner lid 16. The inner lid 16 has a large area and hence leads to a more efficient production of the nano-materials compared with a case of absence of the inner lid 16.

More specifically, according to the second embodiment of the present invention, the powder of the waste tire and the water, for example, as the source material 5 are charged and tightly enclosed in the pressure vessel 2 which may be easily opened, the pressure vessel 2 is heated from the outside and the internal pressure is suddenly decreased so that the water vapor explosion is set off in the pressure vessel 2 and the powdery source material 5 becomes super-fine powders, thereby the nano-carbons are formed. Thereby, nano-carbons can be formed in an atmosphere having a relatively low temperature about 100 - 200 °C. Therefore it is possible to efficiently produce desired nano-carbons by means of such a low-cost equipment without deteriorating properties of the source material.

In the descriptions above, both the apparatuses and the

production methods differ from the first embodiment to the second embodiment, however, the production methods of the first embodiment and the second embodiment can be combined together. Moreover, the production method of the first embodiment and the apparatus
5 of the second embodiment can be combined.

Moreover, it is possible to combine the apparatus according to the first embodiment or the second embodiment and any production method distinct from any of the embodiments. More specifically, heating of the source material 5 is achieved by injecting saturated
10 water vapor or over-heated water vapor. Though, similar to the first embodiment, the water vapor explosion can be set off by injecting the water, the explosion can be set off by decreasing the internal pressure caused by opening the pressure relief device 10. The source material 5 undergoes chain explosions led from
15 breaking equilibrium cause by breaking the over-saturated state. The nano-materials generated in this way are different from the nano-materials generated by the aforementioned water vapor explosion and an example thereof is shown in Fig. 14.

A cylinder type nano-material production apparatus 21
20 according to a third embodiment of the present invention will be described hereinafter with reference to Figs. 4 and 5.

The cylinder type nano-material production apparatus 21 is provided with a cylindrical furnace body 22 and utilized in such a way of being horizontally disposed. The furnace body 22 is
25 provided with flange portions at both ends thereof and end plates 23R and 23L fastened thereto. The end plate 23L shown in the left of drawings is configured to be openable and closable.

On an upper side of the furnace body 22, a pressure gauge 25, a liquid injection nozzle 26 and a pair of safety valve 27
30 are provided. To the right of the upper side of the furnace body 22, a source material charge port 28 is provided. Furthermore, on the right end plate 23R, a temperature sensor 29 for indicating a temperature is provided.

On an outer surface of a lower side of the furnace body 22,
35 at a position corresponding to the source material charge port 28, a heating device 32 housing a heater 30 and covered by a heat insulator 31 is provided. The heating device 32 can be provided

in an inner side of the furnace body 22 so as to heat the source material 5 charged therein. In this case, the heat insulator 31 can be omitted. Moreover, on an inner side, to the somewhat left, of the furnace body 22, a capture device 35 for capturing nano-materials is provided. The capture device 35 is provided with plural capture plates 33, which is made in a circular shape having substantially the same diameter as an inner diameter of the furnace body 22, and plural tie rods 34 for supporting them. The capture device 35 is capable of being taken out and put in the furnace body 22 in a state that the end plate 23 L of the left side is opened. The capture plates 33 are manufactured from, for example, the aforementioned circular plate of the punching metal and such.

Production of nano-materials by utilizing the cylinder type nano-material production apparatus 21 is performed with the following steps. The source material 5 is charged through the source material charge port 28 and the furnace body 22 is tightly closed. Next, as the pressure is appropriately relieved, the temperature and the internal pressure are increased so that saturation and equilibrium are achieved. Next, water is injected into the furnace body 22 through the liquid injection nozzle 26 so that water vapor explosion is set off. By undergoing the water vapor explosion, the nano-materials are produced and adhere to the respective capture plates 33. The nano-materials obtained at the farer sites from the explosion point are smaller in size. Since the plural capture plates 33 are respectively disposed at various distances from the explosion point, various sizes of the nano-materials are obtained depending on the distances.

In a case where the waste tire is applied to the source material 5, it contains small metal powders such as pieces of wires. The metal powders function as nuclei for the nano-carbons. The metal powders attract nano-materials therearound in a case of being heated so as to form lumps thereof, which adhere to the inner wall of the furnace body 22 or the capture device 35. In a case where needle-like metal pieces instead of the metal powders are applied, nano-materials adhere to tips of the metal pieces.

More specifically, it is understood that greater energy of

the water vapor explosion leads to formation of the finer nano-carbons and mediating the metal powders leads to formation of greater amount of nano-materials.

According to the cylinder type nano-material production apparatus 21 of the third embodiment of the present invention, the smaller the nano-materials are, the farer from a site where the source material 5 is charged and the more distant from the explosion point the nano-materials scatter. The plural capture plates 33 respectively capture these nano-materials, thereby particular sizes of the nano-materials can be respectively collected by the capture plates 33.

Next, the source material applied to the present invention will be described hereinafter with reference to Figs. 6 and 7. In these drawings, arrows A indicate flows of process steps.

Initial materials for the source materials are rubbers such as a waster tire, a used belt, an exhausted rubber and such. The initial materials are not limited to these wastes of petroleum products, however, the present invention has an advantage of capability of utilizing the wastes of the petroleum products and it is significant in view of the protection of the environment. The initial materials are charged into a vacuum dry distillation furnace 37 provided with a reflux bath 36. In the vacuum dry distillation furnace 37, the initial materials are thermally cracked about 400 °C at the reflux bath 36 via a safety device 38 and a heat exchanger 39, oil and such are removed therefrom at an oil collector 40 and distilled objects with the metal pieces are collected. Residual carbon reaches 60 %. Oil as a by-product has a good quality and is about 30 %. The remaining about 10 % is metal. One of features is that the collection rate of the oil as the by-product is limited to be about 30 % so as to increase the amount of the residual carbon.

Next, since the carbon black 41 produced by the dry distillation furnace 37 is lumpish, the carbon black 41 is roughly crushed in a rough crusher 42 and wire pieces are removed therefrom by a magnet roll 43.

The carbon black 44 from which the metal pieces are removed is charged into a double rotary kiln 45 and heated from the outside

by the rotary kiln 45 so as to thermally change. As a result, unnecessary substances are oxidized and removed, thereby the carbon black 44 is changed to be insusceptible to further thermal change. Bad smell of the rubber and unnecessary substances are removed.

5 The carbon black 46 treated by the rotary kiln 45 is subject to a fine crusher 48 and a vibrating sieve 49 via a hopper 47 and thereby comes to be a source material 50 so that a grain size thereof is regulated to be not greater than 2 mm.

10 As being understood from the above, according to the method of production of the nano-source-material of the present invention, the residual carbon after collecting the oil, the metal and such from the wastes of the petroleum products such as the waste tire can be used as the source material and it is reprocessed so as to produce the carbon black which is applied to the source material
15 50 for the nano-material. According to the present invention, the waste of the petroleum products such as the waste tire, which do not have any definitive re-use, can be used for production of the nano-carbon, which has a high added value. The present invention enables them to provide the nano-carbons far low in cost
20 than ever before.

Former dry distillation had been achieved at a relatively high temperature about 800 °C mainly in view of how much oil can be recycled. Since the dry distillation of the present invention can be achieved at a far lower temperature of 350-400 °C, the equipments can be made simpler. Moreover, it leads to smaller
25 concern about generation of unfavorable substances such as black smoke and dioxin by incineration.

As described already, nuclei are necessary to capture the carbon clusters which are made super-fine particles and metal powder
30 such as iron powder and steel powder can be added as the nuclei. It should be determined depending on what nano-materials should be produced whether they are added or not.

Although the invention has been described above by reference to certain embodiments of the invention, the invention is not
35 limited to the embodiments described above. Modifications and variations of the embodiments described above will occur to those skilled in the art, in light of the above teachings.

For example, as a method of inducing the water vapor explosion, inflation function can be utilized. More specifically, the water vapor explosion can be induced by providing an inflation valve with a pressure vessel, injecting heated source material under
5 pressure into the inflation valve and emitting it into the pressure vessel at a lower pressure. Nano-materials can be produced by capturing the nano-materials contained in the water vapor explosion according to a method similar to the aforementioned method.

Dimensions of the nano-material production apparatuses of
10 the aforementioned embodiments can be changed. For example, the cite of the capture device can be changed in various ways and then kinds of the nano-materials vary depending thereon. Moreover, further efficient production can be possible. Furthermore, the source material after the reaction may be re-charged into the
15 inflation valve so as to increase yield.

Next, examples of the present invention will be described hereinafter with reference to Figs. 8 through 14. Figs. 6 and 7 shows electron microscopic images of the source material obtained by the aforementioned production method. Fig. 8 shows an example
20 of the nano-tubes obtained from the source material by the nano-material production apparatus 21 according to the third embodiment. This was obtained at an intermediate cite from the explosion point. The nano-tube obtained by the present invention has a feature of not a well-known needle-like shape but a long
25 thread-like shape. It is considered to be caused by that the water vapor has an atmosphere promoting growth of the nano-tubes. Increase in number of water vapor explosion or continuous water vapor explosion leads to production of further longer nano-tubes.

Figs. 9 through 12 are electron microscopic images of obtained
30 nano-materials arranged in an order by one distant from and another nearer to the explosion point. Depending on the cites, various characteristics of nano-materials such as oyster-shell-like, cauliflower-like and gathered-needle-like nano-materials are obtained.

35 Figs. 13 and 14 are examples of nano-materials obtained by water vapor explosion set off by pressure relief. Fabric-like and ball-like nano-materials are respectively obtained.

INDUSTRIAL APPLICABILITY

As being understood from the above description, by utilizing water vapor explosion, nano-materials can be produced at a relatively low temperature about 100-200 °C. Because of the relatively low temperature, the nano-materials can be produced with a simple apparatus, with facility and in a high efficiency. Moreover, various characteristics of nano-materials can be produced by changing cites for capturing the nano-materials. Furthermore, wastes of petroleum products such as waste-tires, used belts and exhausted rubbers can be applied to source materials and it is significant in view of the protection of the environment.

CLAIMS

1. A method of production of a nano-material, the method comprising:
 - 5 mixing a substance which is to be a carbon source and a liquid so as to form a source material;
 - heating the source material in a pressure vessel to around a boiling point so as to be in an equilibrium state;
 - giving a disturbance to the equilibrium state so as to set
 - 10 off a water vapor explosion; and
 - making the nano-materials adhere to an inner wall of the pressure vessel.
2. The method of production of a nano-material of claim 1, wherein:
 - 15 the disturbance is injection of liquid to the heated source material.
3. The method of production of a nano-material of claim 1, wherein:
 - the disturbance is relief of a pressure of the heated source
 - 20 material.
4. The method of production of a nano-material of claim 1, wherein:
 - the disturbance is release of the source material in an over-heated state into the vessel through an inflation valve.
- 25 5. A nano-material production apparatus comprising:
 - a pressure vessel;
 - a heating device for heating a source material disposed in the pressure vessel;
 - an injection device for injecting a liquid into the source
 - 30 material so as to set off a water vapor explosion in the pressure vessel;
 - a capture device disposed in the pressure vessel;
 - a pressure regulation device for regulating a pressure in the pressure vessel; and
 - 35 a control device timely operating the injection device.
6. The nano-material production apparatus of claim 5, wherein:
 - the pressure vessel is an autoclave and the heating device

is configured to heat a portion of the autoclave, the portion receiving the source material, or a whole of the autoclave.

7. The nano-material production apparatus of claim 6, wherein:
a bottom of the autoclave is configured to receive the source

5 material and the capture device is disposed above the bottom so as to partition the autoclave into an upper half and a lower half.

8. The nano-material production apparatus of claim 7, wherein:
the capture device is a plate having plural through holes.

9. The nano-material production apparatus of claim 5, wherein:
10 the pressure vessel is cylindrically shaped and horizontally disposed and comprises an open-and-closable lid on at least one end thereof and plural capture members in rows being capable of being taken out and put in the pressure vessel.

10. A method of production of a source material for production
15 of the nano-material, the method comprising:

dry-distilling a resin at a temperature in a range from 350 to 400 °C;

removing an impurity from a residual substance formed by dry-distilling;

20 oxidation-removing an unnecessary substance from the residual substance; and

regulating a grain size of the residual substance.

11. The method of production of a source material for production of the nano-material of claim 10, the method further comprising:

25 adding a metal powder.