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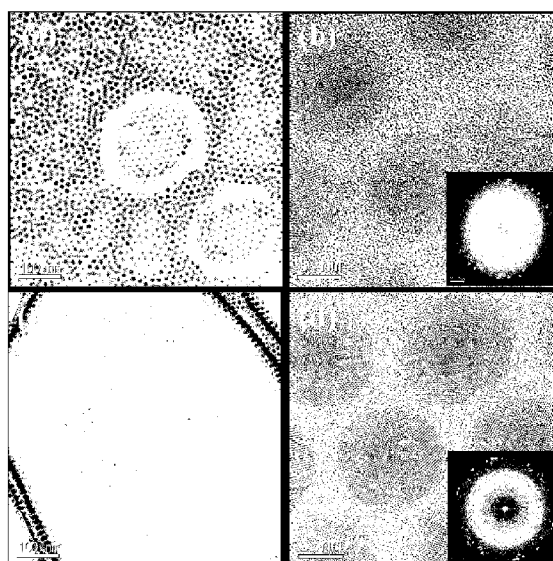
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(54) Title: A METHOD FOR MANUFACTURING ULTRA-THIN CARBON SUPPORTING FILM



(57) Abstract: Disclosed is a method for manufacturing an ultra-thin carbon support film. The method provides the ultra-thin carbon support film used as a specimen for high resolution transmission electron microscope (HRTEM) analysis for analyzing atom structures of nano-particle specimens. The ultra-thin carbon support film is fabricated such that a large-sized holey region is formed in the ultra-thin carbon support film, so that it is possible to obtain images of particles lying on the holey region through the HRTEM analysis while minimizing the size of the support film in the images. The ultra-thin carbon support film has a thinner thickness while maintaining structural and thermal stabilities, so the ultra-thin carbon support film is advantageously used for the HRTEM analysis of specimens.

WO 2007/126165 A1

Description

A METHOD FOR MANUFACTURING ULTRA-THIN CARBON SUPPORTING FILM

Technical Field

- [1] The present invention relates to a method for manufacturing an ultra-thin carbon support film, more particularly to a preparation method for nano-particle specimens, which are used as test samples for a high-resolution transmission electron microscope (HRTEM) and importance and demand of which have become greatly increased with the development of nano-technologies.

[2]

Background Art

- [3] In the process of high-resolution analysis for nano-particle specimens, the image quality greatly depends on the thickness of a specimen support film. In general, the film thickness of a metal mesh grid having a commercial carbon support film thereon is about several tens of nanometers, so that it is difficult to obtain the high-resolution image for the nano-particles due to interference with the amorphous image of the support film. In order to solve the above problem, various studies and research have been performed to fabricate ultra-thin carbon support films, and grids prepared with the ultra-thin carbon support films are now available from the market. However, these ultra-thin carbon support films also present problems because they are too thick to provide a microstructure of nano-scale particles having a size of 10nm or less. In order to perform high-resolution analysis for the nano-scale particles having a size of few nanometers, it is necessary to observe particles lying on a thin film area sustaining in a holy region of the grid having the commercial ultra-thin carbon support film thereon.
- [4] However, it is very difficult to precisely find particles lying on the thin film area having a favorable thickness. Although it is possible to observe the microstructure of the individual particle by finding the particles lying on predetermined positions, a specimen must be separately fabricated in order to obtain two or three-dimensional structure images of the nano-particles, such as a self-assembly behavior of the nano-particles.
- [5] FIG. 1 is a view illustrating an HRTEM image of a nano-particle specimen prepared with a metal mesh grid having a commercial carbon support film thereon. It can be understood from FIG. 1 that the specimen image cannot be accurately obtained due to interference with the carbon support film, which supports the specimen. In addition, a diffractogram, which is obtained as a result of fast Fourier transform for a corresponding image, shows that the signal for the specimen structure seriously interferes

with a diffused amorphous image signal caused by the carbon support film.

[6] FIG. 2 is a view illustrating HRTEM specimens of nano-particles prepared with an ultra-thin carbon support film so as to solve the problem derived from a conventional carbon support film grid. When analyzing nano-particles lying on thin film areas of the carbon support film, which are formed over holes of the carbon support film, if the nano-particles are prepared with the conventional carbon support film, the specimen observation area is too small and the support film lying on the thin film area is too thick, so that it is difficult to obtain the clear high-resolution image. In addition, since the thin film area for supporting the specimen has a limited size, it is difficult to observe the self-assembly behavior of the nano-particles.

[7] The problems occurring in the prior art are as follows:

[8] 1) The commercial ultra-thin carbon support film is so thick that the structure of the nano-particles may not be easily observed by the HRTEM.

[9] 2) The HRTEM specimen employing the commercial ultra-thin carbon support film for high-resolution analysis has a limited observable area and may not allow the support film to have a sufficiently thin thickness.

[10]

Disclosure of Invention

Technical Problem

[11] Therefore, the present invention has been made in view of the above-mentioned problems occurring in the prior art, and it is an object of the present invention to provide a method for manufacturing an ultra-thin carbon support film, wherein the method reduces the thickness of the ultra-thin carbon support film, thereby minimizing interference with the specimen image and allowing the ultra-thin carbon support film to have mechanical and thermal stabilities.

[12]

Technical Solution

[13] In order to accomplish the above object, according to the present invention, there is provided a method for manufacturing an ultra-thin carbon support film used for high resolution transmission electron microscope (HRTEM) analysis, the method comprising the steps of: placing a clean hydrophobic slide glass in a refrigerator, in a freezer or on an ice pack and exposing the slide glass to an atmosphere by picking up the slide glass using tweezers, thereby forming droplets in the slide glass; immersing the chloroform solution mixed with a formvar or a butvar solution, taking out the slide glass after several seconds, and then drying the slide glass by vertically installing the slide glass on a ground while interposing a filter paper between the slide glass and the ground; floating a polymer film formed on the slide glass on a surface of distilled

water by means of surface tension, and then placing a Cu grid, which is a metal mesh grid, on the polymer film floating on the surface of distilled water; and taking the polymer film out of the distilled water by means of a hydrophobic supporter including a paraffin film, and coating carbon on the polymer film.

[14]

Advantageous Effects

[15] As can be seen from the foregoing, in comparison with the specimen prepared with the conventional ultra-carbon support film, the HRTEM specimen prepared with the ultra-thin carbon support film fabricated according to the method of the present invention represents following advantages.

[16] 1) The ultra-carbon support film fabricated according to the present invention has a thickness 1.6 times thinner than that of the conventional ultra-carbon support film, so the ultra-carbon support film fabricated according to the present invention can be advantageously used for high resolution structure analysis.

[17] 2) The specimen observable area in the ultra-thin carbon support film fabricated according to the present invention is about $67 \pm 7.6\%$, which is about twice as large as the specimen observable area in the conventional ultra-thin carbon support film ($32.9.3\%$), so that it is easy to find the specimen suitable for observation and two-dimensional or three-dimensional self-assembly of the nano-particles can be observed in one specimen, improving the working efficiency.

[18] 3) The thickness deviation of the ultra-thin carbon support film fabricated according to the present invention is within $\pm 3\%$. The above thickness deviation is similar to that of the conventional ultra-thin carbon support film. However, since the thickness of the ultra-thin carbon support film fabricated according to the present invention is thinner than that of the conventional ultra-thin carbon support film, the absolute value of thickness deviation of the ultra-thin carbon support film fabricated according to the present invention may be smaller than that of the conventional ultra-thin carbon support film.

[19] While this invention has been described in connection with what is presently considered to be the most practical and preferred embodiment, it is to be understood that the invention is not limited to the disclosed embodiment and the drawings, but, on the contrary, it is intended to cover various modifications and variations within the spirit and scope of the appended claims.

[20]

Brief Description of the Drawings

[21] FIG. 1 is a view illustrating an HRTEM image (left) of a nano-particle specimen prepared with a metal mesh grid having a commercial carbon support film thereon, and

a fast Fourier transform image (right) obtained as a result of fast Fourier transform for the HRTEM image;

[22] FIG. 2 is a view illustrating HRTEM specimens of nano-particles prepared with an ultra-thin carbon support film so as to solve the problem derived from a conventional carbon support film;

[23] FIG. 3 is a view illustrating HRTEM images, in which (a) shows an HRTEM image of a nano-particle specimen prepared with a commercial ultra-thin carbon support film, (c) shows an HRTEM image of an oxidized iron nano-particle specimen prepared with an ultra-thin carbon support film according to the present invention, (b) shows an HRTEM image obtained from the specimen observable area marked in (a) by an arrow, and (d) shows an HRTEM image obtained from the specimen observable area marked in (c) by an arrow;

[24] FIG. 4 is a view for comparing the percentage of the observable area between a commercial ultra-thin carbon support film and an ultra-thin carbon support film fabricated according to the present invention;

[25] FIG. 5 is a view illustrating histogram results between a conventional ultra-thin carbon support film and an ultra-thin carbon support film fabricated according to the present invention;

[26] FIG. 6 is a view illustrating an HRTEM image and a graph, in which (a) shows a thickness map for an ultra-thin carbon support film fabricated according to the present invention, and (b) shows thickness deviation between a conventional ultra-thin carbon support film and an ultra-thin carbon support film fabricated according to the present invention; and

[27] FIG. 7 is a view illustrating HRTEM images, in which (a) is an HRTEM image of nano-particles prepared with an ultra-thin carbon support film, (b) is a three-dimensional self-assembly behavior of nano-particles lying on an ultra-thin carbon support film, (c) is a diffractogram obtained as a result of fast Fourier transform for an image of the three-dimensional self-assembly behavior of nano-particles, and (d) is a model schematically representing the self-assembly behavior of nano-particles stacked with a hexagonal symmetry structure in the diffractogram.

[28]

Mode for the Invention

[29] Hereinafter, a method for manufacturing an ultra-thin carbon support film according to the present invention will be described with reference to accompanying drawings.

[30] In addition, the ultra-thin carbon support film manufactured by the method of the present invention will be explained in comparison with an ultra-thin carbon film on Cu grid (300mesh) (hereinafter, referred to as a commercial or conventional ultra-thin

carbon film)", which is used for high-resolution analysis and is available from Ted Pella (U.S.).

[31] (1) Preparation of ultra-thin carbon support film

[32] A conventional method of manufacturing the carbon support film having holes is modified to form water droplet on a hydrophobic glass slide in order to form large holes on the carbon support film and to form thin films over the holes. The hydrophobic glass slide, which has been previously cooled in the refrigerator or on the ice pack, is exposed to an atmosphere. The size and distribution of the water droplets formed on the hydrophobic glass slide can be adjusted by controlling the detention time of the hydrophobic glass slide in the atmosphere. In general, the hydrophobic glass slide is exposed to the atmosphere within a short period of time. However, according to the present invention, water droplets having sizes suitable for forming the ultra-thin carbon support film can be formed by exposing the hydrophobic glass slide to the atmosphere for 5 to 60 second seconds under the conditions of 20~28°C room temperature and 30~70% humidity. Although it is necessary to control process conditions, such as the ambient temperature, humidity, and convection current, in order to precisely control the size of the water droplets, since the main idea of the present invention is to utilize the thin support film lying over the holes having relatively large sizes, the present invention is not sensitive to the above process conditions. A chloroform solution mixed with a solution of 0.25~0.5% formvar is used so as to form the support film. In addition, a carbon coating process has been performed by using a carbon coater (DV-502A, Denton Vacuum) under the vacuum atmosphere of 1×10^{-5} to 5×10^{-5} torr while heating a carbon bar for 20 to 60 seconds by applying current of 15A or less to the carbon bar.

[33] Details of the basic procedure for preparing the ultra-thin support film are described below.

[34] 1) A hydrophobic treated slide glass, which is carefully cleaned with water and dried with a piece of cloth, is kept in the cool area, such as in the refrigerator or on the ice pack.

[35] 2) The slide glass is picked up by a pair of tweezers and exposed to the atmosphere. The size of the water droplet becomes increased as time lapses. In general, a favourable droplet size for making the ultra-thin carbon support film over the holes is 20 μ m or less in mean diameter. The detention time for producing a suitable size of water droplets is ranged from 5 to 60 under the conditions of the room temperature of about 20 to 28°C and indoor humidity of about 30 to 70%.

[36] 3) The slide glass formed with the water droplets is immersed into the chloroform solution mixed with a solution of 0.25~0.5% formvar (or butvar), and then is taken out after 2 to 10 seconds. After that, the slide glass is dried by vertically installing the slide

glass on the ground while interposing a filter paper therebetween.

[37] 4) A polymer film formed on the slide glass consists of a holey region including holes having sizes of few micrometers and a thin film region including a thin polymer film without holes. The polymer film formed on the slide glass is floated on a surface of distilled water by means of surface tension. Then, a Cu grid, which is a metal mesh grid, is placed on the polymer film floating on the surface of distilled water. After that, the polymer film is taken out of the distilled water by means of a hydrophobic supporter, such as a paraffin film, and then carbon is coated on the polymer film.

[38] 5) The carbon coating process is performed by means of typical carbon/gold deposition equipment under the vacuum atmosphere of 1×10^{-5} to 5×10^{-5} torr while resistance-heating a carbon bar for 20 to 60 seconds by applying current of 5A to 15A to the carbon bar. The carbon coating thickness is controlled on the basis of color change of a white filter paper from white to light gray color while the carbon coating is being performed by inserting the white filter paper into the carbon coater. A worker can easily recognize the color change of the white filter paper under the above process conditions.

[39] In this manner, the metal mesh grid having the ultra-thin carbon support film thereon can be fabricated. Such a metal mesh grid can be used as a nano-particle specimen for HRTEM analysis. Meanwhile, it is possible to remove the formvar or butvar polymer film by exposing the slide glass to chloroform steam. However, performance of the product equipped with the thin-carbon support film according to the present invention may be higher than that of the product equipped with the conventional thin-carbon support film, even if the polymer film remains on the product.

[40] (2) Property estimation of the ultra-thin carbon support film

[41] Oxidized iron nano-particles having magnetic properties were used as test samples in the performance test for the specimen support film. In addition, overall aspects of the ultra-thin carbon support film have been acquired by using an HVEM (JEM-ARM1300S, JEOL). A post column image filter (HV-GIF, Gatan) mounted in the HVEM (JEM-ARM1300S, JEOL) was used in order to analyze the results of EELS (electron energy loss spectroscopy) for thickness deviation of the carbon support film and to analyze the histogram representing the image quality. In addition, properties of the product equipped with the ultra-thin carbon support film according to the present invention were compared with those of the product equipped with the conventional ultra-thin carbon support film.

[42] In order to compare the ultra-thin carbon support film fabricated according to the present invention with the commercial carbon support film, a specimen is prepared by using oxidized iron nano-particles, and then the specimen is observed by means of the HVEM. FIG. 3 shows the observation result. FIG. 3(a) shows an HRTEM image of

nano-particles prepared with the commercial ultra-thin carbon support film, and FIG. 3(c) shows an HRTEM image of oxidized iron nano-particles prepared with the ultra-thin carbon support film according to the present invention. When comparing FIG. 3(b) with FIG. 3(d), it can be understood that the nano-particles prepared with the ultra-thin carbon support film according to the present invention can obtain more clear HRTEM images as compared with those of nano-particles prepared with the conventional ultra-thin carbon support film, since interference caused by the support film can be reduced in the HRTEM images of the nano-particles prepared with the ultra-thin carbon support film according to the present invention. The EELS measurement has been performed with respect to the corresponding observable area so as to compare the relative film thickness (t/λ) of both support films. As a result, it is determined that the ultra-thin carbon film according to the present invention is about 1.6 times thinner than the conventional ultra-thin carbon film.

[43] As discussed with reference to FIG. 3, the conventional ultra-thin carbon film is sustained in the small-sized holey region representing irregular size distribution. On the contrary, the ultra-thin carbon support film fabricated according to the present invention can be effectively sustained in a large-sized holey region representing uniform size distribution, so that the ultra-thin carbon support film is suitable for high-resolution analysis of nano-particle specimens. FIG. 4 shows comparison results for specimen observable areas between the conventional ultra-thin carbon support film and the ultra-thin carbon support film fabricated according to the present invention. The specimen observable area in the ultra-thin carbon support film fabricated according to the present invention is about $67 \pm 7.6\%$, which is about twice as large as the specimen observable area in the conventional ultra-thin carbon support film ($32 \pm 9.3\%$).

[44] The image quality of the specimen can be analyzed based on the histogram representing the brightness and contrast. As the width of the histogram becomes narrow, the contrast becomes high. In addition, the brightness is improved as the size of the histogram increases. FIG. 5 shows the histogram results of a conventional ultra-thin carbon support film and an ultra-thin carbon support film fabricated according to the present invention. It can be understood from FIG. 5 that the image of the specimen prepared with the ultra-thin carbon support film fabricated according to the present invention represents the contrast and brightness higher than those of the specimen prepared with the conventional ultra-thin carbon support film. That is, since the ultra-thin carbon support film fabricated according to the present invention has a thickness smaller than that of the conventional ultra-thin carbon support film, the structural image of the specimen can be effectively obtained.

[45] In high resolution analysis for nano-particles, one of parameters deteriorating the specimen image is thickness non-uniformity. The support film may cause non-

uniformity to the high resolution image. In general, the conventional ultra carbon support film represents thickness deviation of about $\pm 3\%$. Such thickness deviation of the support film can be analyzed through electron energy loss spectroscopy (EELS). In detail, after obtaining the zero-loss image using a slit having energy of about 10eV, the slit is shifted into a first plasmon-loss peak position, thereby obtaining a plasmon-loss image. After that, an image representing intensity of relative thicknesses (t/λ) is obtained on the basis of equation $t/\lambda = -\ln(1 + I_o/I_p)$. In addition, thickness deviation in the specimen area can be obtained based on the contrast of the image.

[46] FIG. 6 is a view illustrating an HRTEM image and a graph, in which (a) shows a thickness map for an ultra-thin carbon support film fabricated according to the present invention, and (b) shows thickness deviation between a conventional ultra-thin carbon support film and an ultra-thin carbon support film fabricated according to the present invention, which is obtained by analyzing the horizontal intensity profile from the thickness map. In both support films, the thickness deviation is within $\pm 3\%$. Referring to FIG. 6b, it seems that the ultra-thin carbon support film fabricated according to the present invention represents serious thickness deviation as a function of the distance. However, in practice, since the thickness of the ultra-thin carbon support film fabricated according to the present invention is 1.6 times thinner than that of the conventional ultra-thin carbon support film, the absolute value of thickness deviation of the ultra-thin carbon support film fabricated according to the present invention may be smaller than that of the conventional ultra-thin carbon support film.

[47] In order to realize high resolution analysis for nano-particles having a size of few nanometers and to obtain the image in relation to the two-dimensional or three-dimensional self-assembly behavior, the specimen support film must have thin and uniform thickness and large size. Such a specimen support film is easily fabricated by means of the manufacturing method according to the present invention. In addition, it can be understood through the EELS and the image analysis technique that the ultra-thin carbon support film fabricated according to the present invention represents superior characteristics as compared with those of the conventional ultra-thin carbon support film.

[48] FIG. 7a is an HRTEM image of an ultra-thin carbon support film fabricated according to the present invention, in which oxidized iron nano-particles are lying on the ultra-thin carbon support film. The holy region where the ultra-carbon support film is formed has the thin thickness and large area, so that the nano-particles can be effectively aligned on the holy region. Conventionally, two specimens must be separately prepared by using two specimen support films in order to perform two types of work, causing inconvenience. However, the HRTEM specimen prepared with the ultra-thin carbon support film fabricated according to the present invention may allow

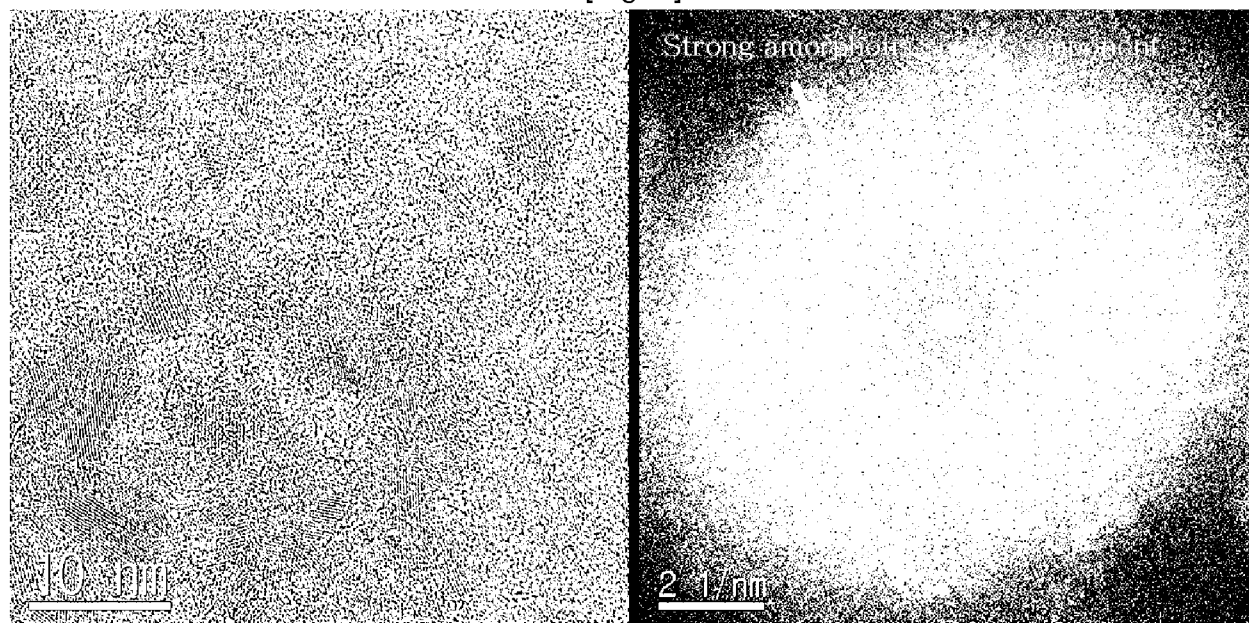
the user to observe the high resolution structural image for each particle and the assembly structure of the particles from one specimen, so the working efficiency can be significantly improved. For instance, FIG. 7b shows a three-dimensional self-assembly behavior of magnetic nano-particles lying on an ultra-thin carbon support film. Referring to FIG. 7c representing a diffractogram obtained as a result of fast Fourier transform, the magnetic nano-particles are effectively stacked on the ultra-thin carbon support film with a hexagonal symmetry structure. The magnetic nano-particles stacked on the ultra-thin carbon support film can be schematically represented in the form of a model as shown in FIG. 7d. Conventionally, a carbon support film having no holes must be formed on a metal mesh grid in order to obtain a self-assembly image for the nano-particles.

[49]

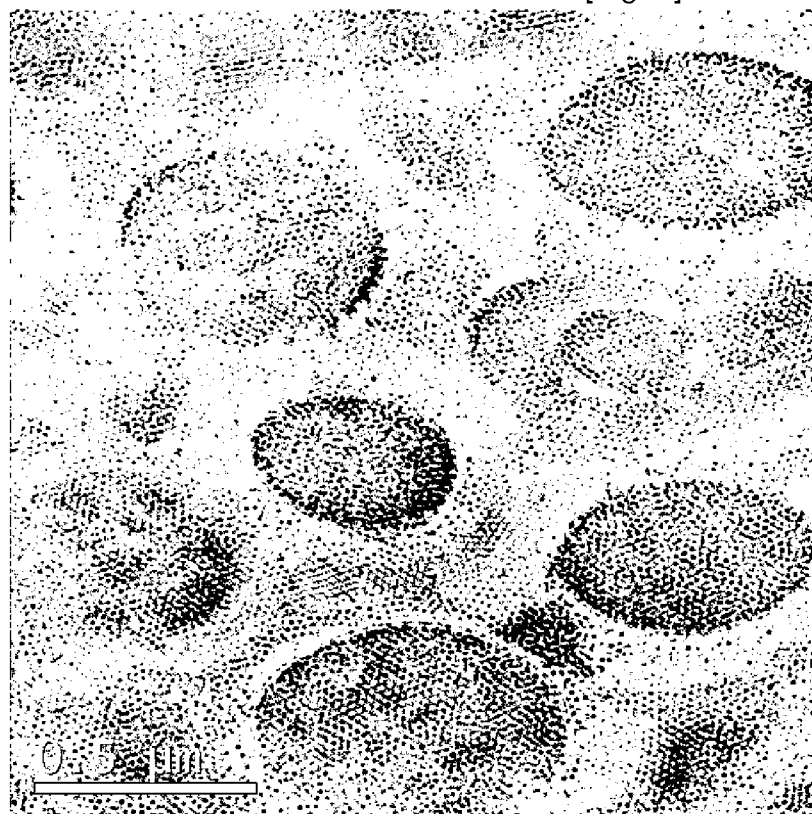
Claims

- [1] A method for manufacturing an ultra-thin carbon support film used for high resolution transmission electron microscope (HRTEM) analysis, the method comprising the steps of:
- 1) placing a clean hydrophobic slide glass in a refrigerator, in a freezer or on an ice pack and exposing the slide glass to an atmosphere by picking up the slide glass using tweezers, thereby forming droplets in the slide glass;
 - 2) immersing the slide glass formed with the droplets into a chloroform solution mixed with a formvar or a butvar solution, taking out the slide glass after several seconds, and then drying the slide glass by vertically installing the slide glass on a ground while interposing a filter paper between the slide glass and the ground;
 - 3) floating a polymer film formed on the slide glass on a surface of distilled water by means of surface tension, and then placing a Cu grid, which is a metal mesh grid, on the polymer film floating on the surface of distilled water; and
 - 4) taking the polymer film out of the distilled water by means of a hydrophobic supporter including a paraffin film, and coating carbon on the polymer film.
- [2] The method as claimed in claim 1, wherein, in step 1), the slide glass is exposed to the atmosphere for 5 to 60 seconds under a room temperature of about 20 to 28°C and an indoor humidity of about 30 to 70% in such a manner that the droplet has a diameter equal to or less than 2 μ .
- [3] The method as claimed in claim 1, wherein, in step 2), the chloroform solution contains 0.25 to 0.5% of the formvar or butvar solution.
- [4] The method as claimed in claim 1, wherein, in step 4), the carbon coating process is performed by means of typical carbon/gold deposition equipment under a vacuum atmosphere of 1×10^{-5} to 5×10^{-5} torr while resistance-heating a carbon bar for 20 to 60 seconds by applying current of 5A to 15A to the carbon bar.
- [5] The method as claimed in claim 4, wherein a carbon coating thickness is controlled on a basis of color change of a white filter paper from white to light gray color while the carbon coating is being performed by inserting the white filter paper into a carbon coater.

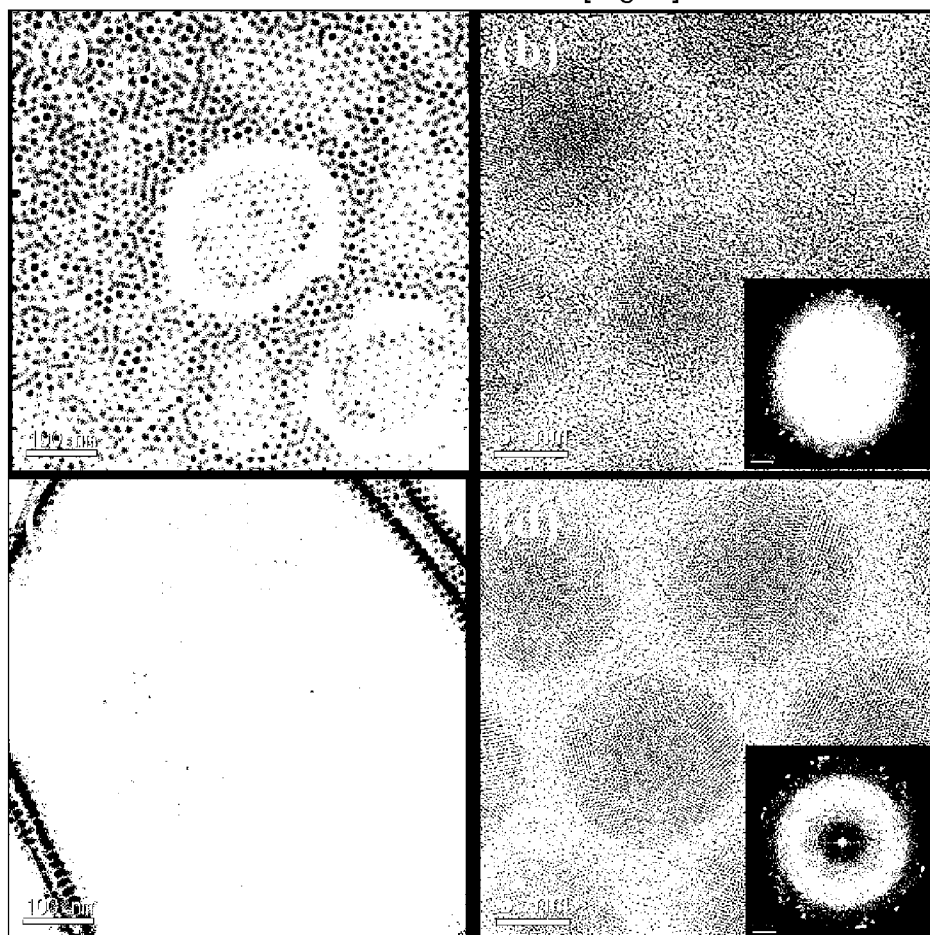
[Fig. 1]



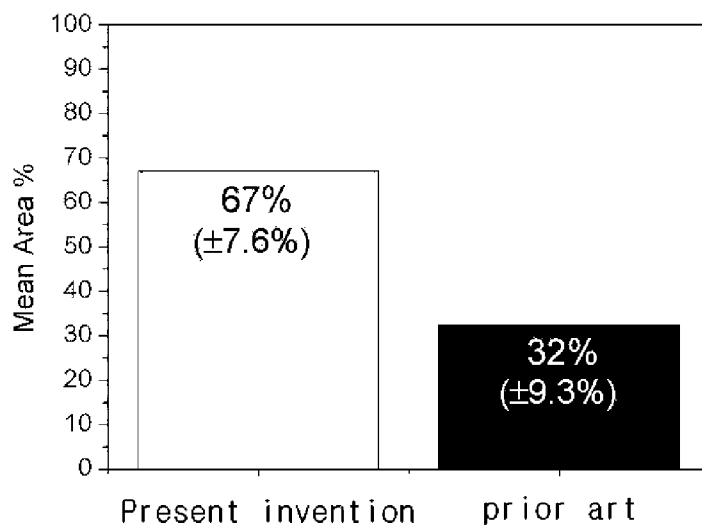
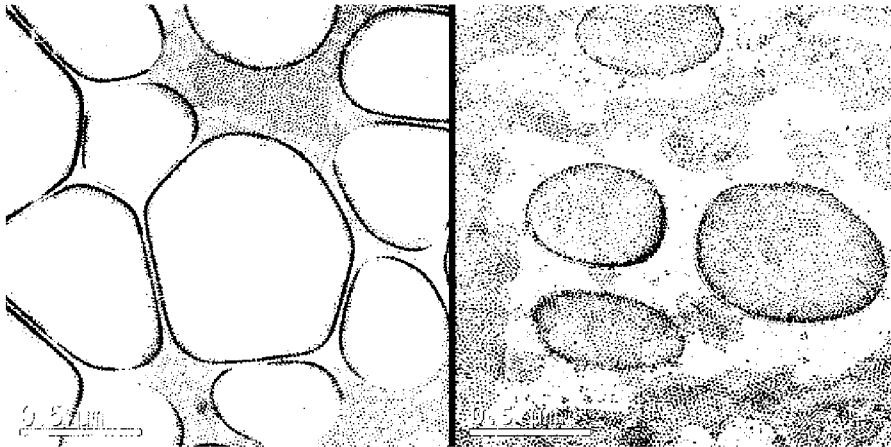
[Fig. 2]



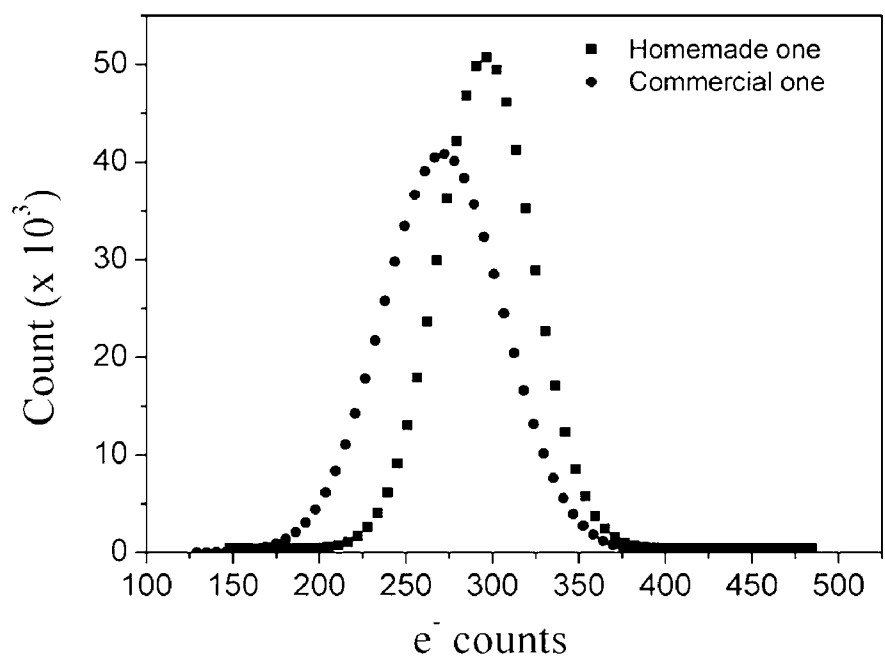
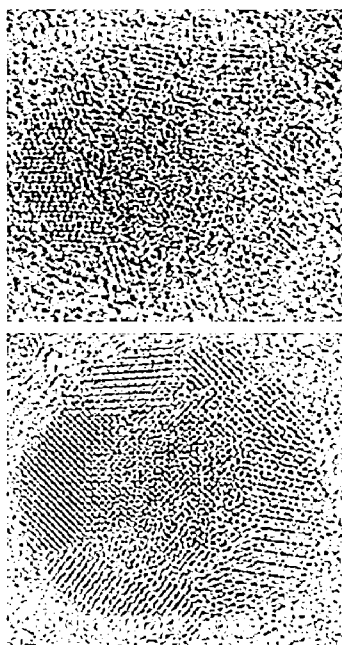
[Fig. 3]

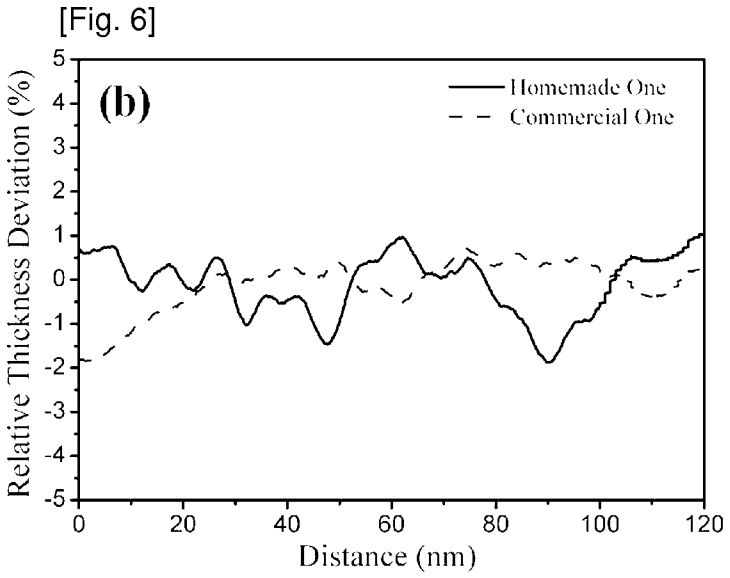
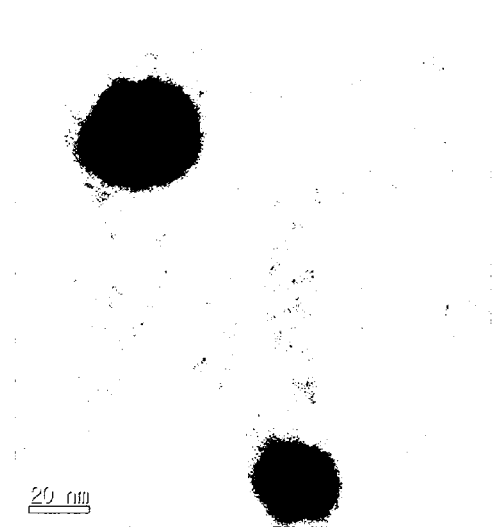


[Fig. 4]

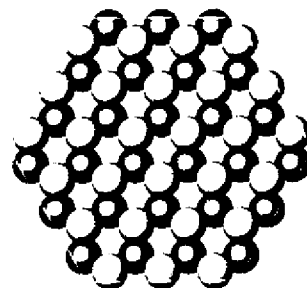
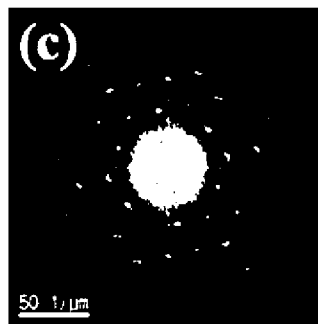
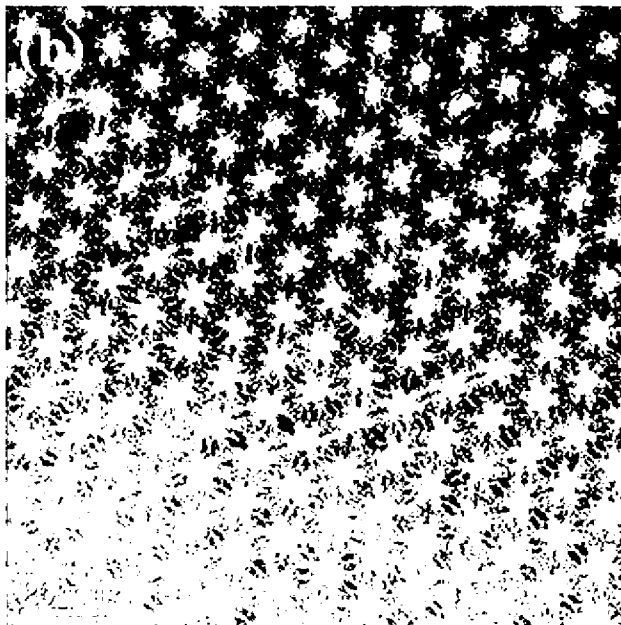
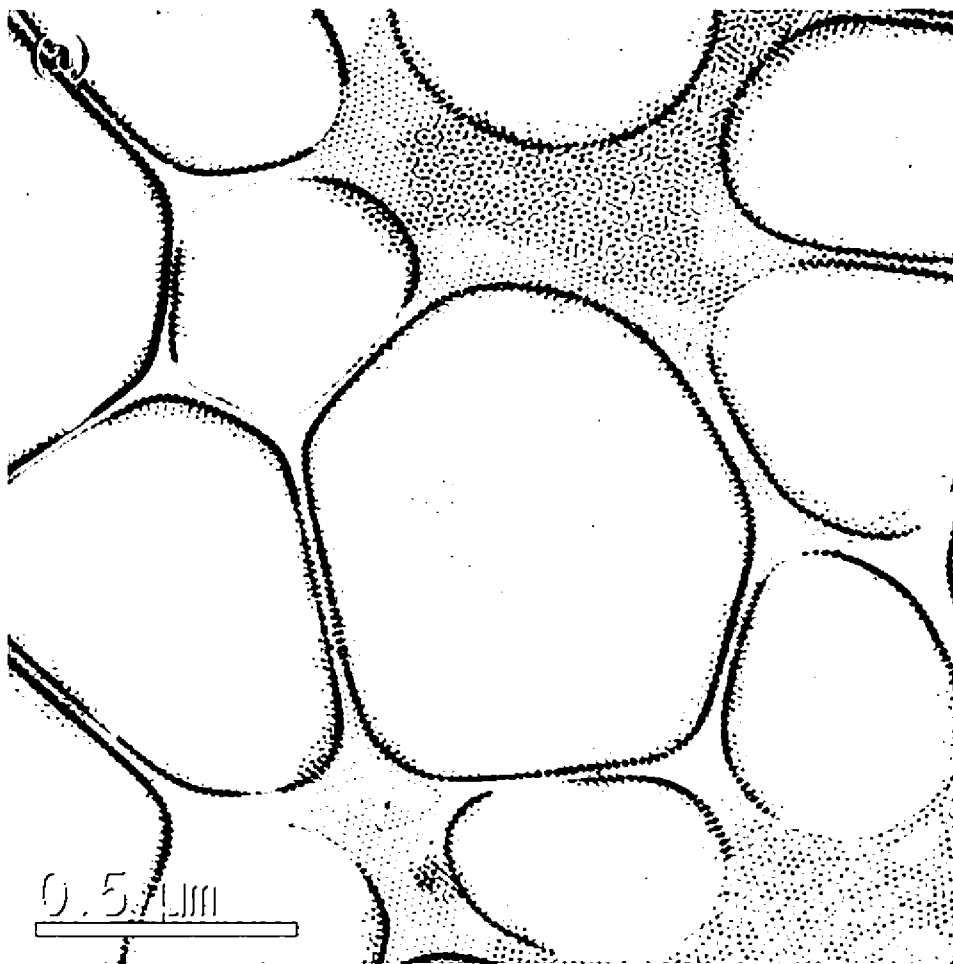


[Fig. 5]





[Fig. 7]



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2006/001613**A. CLASSIFICATION OF SUBJECT MATTER****G01N 27/12(2006.01)i**

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

G01N27/12 IPC8

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
KR, JP as above IPCElectronic data base consulted during the international search (name of data base and, where practicable, search terms used)
KIPONET, "electron", "beam", "mesh", "grid", "membrane"**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US6300631B1 (Shofner) 9 OCT 2001 see the entire documents	1-5
A	US 4163900A (Warren) 7 AUG 1979 see the entire documents	1-5
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