(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 1 839 784 A1

(12) EUROPEAN PATENT APPLICATION
published in accordance with Art. 158(3) EPC

(43) Date of publication:
03.10.2007 Bulletin 2007/40

(21) Application number: **05814736.4**

(22) Date of filing: **09.12.2005**

(51) Int Cl.:
B22F 9/24 *(2006.01)* ***B22F 1/00*** *(2006.01)*
H01B 1/22 *(2006.01)* ***H01B 5/00*** *(2006.01)*

(86) International application number:
PCT/JP2005/022623

(87) International publication number:
WO 2006/062186 (15.06.2006 Gazette 2006/24)

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IS IT LI LT LU LV MC NL PL PT RO SE SI SK TR

(30) Priority: **10.12.2004 JP 2004357836**

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(54) NICKEL POWDER, PROCESS FOR PRODUCING THE SAME, AND CONDUCTIVE PASTE

(57) It is an object of the present invention to provide a nickel powder which is fine, has a narrow particle size distribution, and has a low content or deposit of impurities, such as carbon. It is a further obj ect of the present invention to provide a conductive paste which uses the nickel powder. To achieve these objects, a method of producing a nickel powder in which a reaction solution comprising a nickel salt, a polyol and a noble metal catalyst is heated to a reduction temperature is employed. In the method, nickel ion in the reaction solution are reduced while keeping the reduction temperature, wherein the reduction temperature is from 150 to 210 deg. C, and is 150 to 10 deg. C lower than a boiling point of the polyol. A method of producing a nickel powder is employed in which the reaction solution further comprises a dispersing agent. In addition, a nickel powder produced by the above described method and a conductive paste which contains the nickel powder are provided.

Printed by Jouve, 75001 PARIS (FR)

Description

Technical Field

[0001] The present invention relates to a nickel powder, a production method thereof and a conductive paste. The present invention specifically relates to a nickel powder which can be used, for example, as a raw material for a nickel paste used in the formation of an internal electrode of a multilayer ceramic capacitor and a production method thereof, and a conductive paste which uses the nickel powder.

Background Art

[0002] Nickel powder is used in various applications, such as for forming nickel-powder-containing conductive pastes and various electrodes and circuits. Specifically, nickel is commonly used as an internal electrode in multilayer ceramic capacitors (MLCC), where the internal electrode is obtained by coating a nickel-powder-containing conductive paste on a ceramic dielectric or the like, followed by firing.

[0003] A method of producing such a nickel powder is described in, for example, Patent Document 1, which describes a reduction method in which a solid compound of hydroxide or the like of nickel or the like is suspended in a polyol or polyol mixture which is liquid at a reaction temperature, the suspension is then heated to be a temperature of at least 85 deg. C to reduce the solid compound by the polyol, and the formed metal precipitate is separated. With this method, a nickel powder can be obtained easily and economically.

[0004] [Patent Document 1] Japanese Patent Laid-Open No. 59-173206 (page 1)

Disclosure of the Invention

[0005] However, recently, due to demands for miniaturization and increasing of capacity of MLCCs, the need has arisen to make internal electrodes thinner and the surface of the electrodes smoother. To achieve such purposes, size reduction and a narrow particle size distribution are required for nickel powder. In addition, due to demands for good conductivity of internal electrodes, a nickel powder having a low content or deposit of impurities, such as carbon is required. However, the nickel powder which can be obtained from the method described in Patent Document 1 suffers from the problems that particle size reduction and narrow particle size distribution are insufficient, and that a large amount of organic substances tends to deposit to the surface of the nickel powder.

[0006] Accordingly, it is an object of the present invention to provide a nickel powder which is fine, has a narrow particle size distribution, and has a low content or deposit of impurities, such as carbon. It is a further object of the present invention to provide a conductive paste in which the nickel powder is used.

[0007] In view of the above situations, as a result of extensive investigations carried out by the present inventors, the present invention was completed through the discovery that, in a method of producing a nickel powder in which a reaction solution comprising a nickel salt, a polyol and a noble metal catalyst is heated to a reduction temperature and nickel ion in the reaction solution are reduced while keeping the reduction temperature, the above-described objects can be achieved when the reduction temperature is within a specified range.

Method of producing a nickel powder according to the present invention:

[0008] The method of producing a nickel powder according to the present invention is a method of producing a nickel powder in which a reaction solution comprising a nickel salt, a polyol and a noble metal catalyst is heated to a reduction temperature and nickel ion in the reaction solution are reduced while keeping the reduction temperature, characterized in that the reduction temperature is from 150 to 210 deg. C, and is 150 to 10 deg. C lower than a boiling point of the polyol.

[0009] In the method of producing a nickel powder according to the present invention, the reaction solution preferably further comprises a dispersing agent.

Nickel powder according to the present invention:

[0010] The nickel powder according to the present invention is characterized in that it is a powder produced by the above-described method.

[0011] Further, the nickel powder according to the present invention is characterized by having average particle size obtained by an image-analysis of 0.02 to 0.2 micron meter.

[0012] Further, the nickel powder according to the present invention is characterized by having an average particle size D_{50} of 0.1 to 0.5 micron meter.

[0013] Further, the nickel powder according to the present invention is characterized by having a maximum particle size D_{max} of not greater than 0.7 micron meter.

[0014] Further, the nickel powder according to the present invention is characterized by having a carbon content of not greater than 0.6% by weight.

Conductive paste according to the present invention:

[0015] The conductive paste according to the present invention is characterized by comprising any of the above-described nickel powders.

[0016] The nickel powder according to the present invention or the nickel powder obtained by the production method according to the present invention is fine, has a narrow particle size distribution, and has a low content or deposit of impurities, such as carbon. Further, since the conductive paste according to the present invention uses the nickel powder according to the present invention, a nickel film which is obtained by firing the conductive paste can be made thinner and the surface of the nickel film can be made smoother. As a result, for example, if the conductive paste according to the present invention was used, an internal electrode of a MLCC can be made thinner, and the electrode surface can be made smoother, then smaller size and higher capacity of MLCC can be achieved.

Brief Description of the Drawings

[0017]

Figure 1 is a scanning electron microscope photograph of the nickel powder of Example 1;
Figure 2 is a graph showing the particle size distribution of the nickel powder of Example 1;
Figure 3 is a scanning electron microscope photograph of the nickel powder of Comparative Example 1; and
Figure 4 is a graph showing the particle size distribution of the nickel powder of Comparative Example 1.

Best Mode for Carrying Out the Invention

Method of producing the nickel powder according to the present invention:

[0018] In the method of producing the nickel powder according to the present invention, a reaction solution comprising a nickel salt, a polyol and a noble metal catalyst is heated within the specified temperature range and nickel salt in the reaction solution are reduced while keeping a temperature within the specified temperature range.

[0019] The nickel salt used in the present invention are not especially limited, and examples may be nickel hydroxide, nickel sulfate, nickel nitrate, nickel chloride, nickel bromide, and nickel acetate. Among these examples, nickel hydroxide is preferable, because it does not contain elements such as sulfur, carbon, nitrogen, which may adversely affect operation of the MLCC if contained in an internal electrode of a MLCC. In the present invention, the nickel salt can be used alone or in combination of two or more thereof.

[0020] The polyol used in the present invention is a substance which has a hydrocarbon chain and a plurality of hydroxyl groups. Examples of such a polyol may be at least one selected from the group consisting of ethylene glycol (boiling point of 197 deg. C), diethylene glycol (boiling point of 245 deg. C), triethylene glycol (boiling point of 278 deg. C), tetraethylene glycol (boiling point of 327 deg. C), 1,2-propanediol (boiling point of 188 deg. C), dipropyleneglycol (boiling point of 232 deg. C), 1,2-butanediol (boiling point of 193 deg. C), 1,3-butanediol (boiling point of 208 deg. C), 1,4-butanediol (boiling point of 235 deg. C), 2,3-butanediol (boiling point of 177 deg. C), 1,5-pentanediol (boiling point of 239 deg. C), and polyethylene glycol. Among these examples, ethylene glycol is preferred by excellent handling properties, low boiling point and liquid at ordinary temperatures. The polyol used in the present invention has both functions, reducing agent to the nickel salt and a solvent.

[0021] The noble metal catalysts used in the present invention promote the reduction reaction of the nickel salt by the polyol in the reaction solution. Examples maybe palladium compounds such as palladium chloride, palladium nitrate, palladium acetate, palladium ammonium chloride; silver compounds such as silver nitrate, silver lactate, silver oxide, silver sulfate, silver cyclohexanate, silver acetate; platinum compounds such as chloroplatinic acid, potassium chloroplatinate, sodium chloroplatinate; and gold compounds such as chloroauric acid, sodium chloroaurate. Among these examples, palladium nitrate, palladium acetate, silver nitrate or silver acetate is preferable, because the purity of the obtained nickel powder tends to be higher and production costs can be lower. The catalyst can be used as the above-described compound as it is, or as solution.

[0022] The reaction solution according to the present invention contains the above-described nickel salt, polyol and noble metal catalysts. The reaction solution may be prepared by, for example, putting a nickel salt, polyol and noble metal catalysts into water, followed by stirring tomix the resultant solution. If the noble metal catalysts such as in the case of palladium nitrate or the like was an aqueous solution, the reaction solution can be prepared by just mixing the nickel salt, polyol and noble metal catalysts without additional water. The addition order or mixing method in mixing the nickel salt, polyol and noble metal catalysts, for the reaction solution is not especially restricted. For example, the nickel salt, polyol and noble metal catalysts, and a below-described dispersing agent if necessary, can be pre-mixed to prepare a slurry, and the slurry can be mixed with the remaining polyol to prepare the reaction solution.

[0023] It is preferable for the reaction solution to further contain a dispersing agent if necessary, because the obtained nickel powder tends to be finer and the particle size distribution tends to be narrower. Examples of the dispersing agents used in the present invention may be nitrogen-containing organic compounds such as polyvinylpyrrolidone, polyethyleneimine, polyacrylamide, poly (2-methyl-2-oxazoline); and polyvinyl alcohol. Among these examples, polyvinylpyrrolidone is preferable, because the obtained nickel powder tends to have a narrow particle size distribution. In the present invention, the dispersing agents can be used alone or in combination of two or more thereof.

[0024] In the present invention, a nickel powder is produced by heating the reaction solution to a reduction temperature and reducing the nickel salt in the reaction solution while keeping the reduction temperature. In the present invention, the reduction temperature is within a temperature range satisfying two temperature ranges defined from different viewpoints. In the following, the temperature range defined from a first viewpoint is referred to as first temperature range, and the temperature range defined from a second viewpoint is referred to as second temperature range.

[0025] The reduction temperature has a first temperature range of 150 to 210 deg. C, and preferably 150 to 200 deg. C. If the reduction temperature was within this range, the reduction reaction finishes quickly and the nickel powder obtained is less likely to contain impurities or less likely to deposit impurities onto nickel powder after finishing the reaction, so it is preferable.

[0026] On the other hand, if the reduction temperature was below 150 deg. C, the reduction reaction tends to become very slow, soitisnotpreferable. Further, if the reduction temperature was over 210 deg. C, coarse particles tend to form, and the product obtained from the reduction reaction tends to contain a carbon to be a nickel carbide powder, so it is not preferable .

[0027] The reduction temperature also has a second temperature range of 150 to 10 deg. C lower, preferably 100 to 20 deg. C lower, and more preferably 80 to 30 deg. C lower than the boiling point of the polyol. If the reduction temperature was within this range, the obtained nickel powder might be less likely to form coarse particles or agglomerate, and deposition of the organic compounds which are presumed to be byproducts from reaction of the polyol to the surface of the nickel powder can be prevented, so it is preferable.

[0028] On the other hand, if the reduction temperature was 150 deg. C lower than the boiling point of the polyol, the reduction reaction may hardly proceed, so it is not preferable. Further, if the reduction temperature was 10 deg. C lower than the boiling point of the polyol, organic compounds which are presumed to be byproducts from reaction of the polyol tend to deposit to the surface of the nickel powder, so it is not preferable.

[0029] In the present invention, if the reduction temperature satisfied both the first temperature range and the second temperature range, the obtained nickel powder is fine, has a narrow particle size distribution, and has a low content or deposit of impurities, such as carbon.

[0030] The maintaining time of the reaction solution at the above-described reduction temperature cannot be categorically specified, because the appropriate time depends on the reaction solution composition and reduction temperature. However, the maintaining time can be from 1 to 20 hours, and preferably 2 to 15 hours. If the maintaining time of the reaction solution at the above-described reduction temperature was within this range, particle growth of the nickel powder in the system is almost uniform, because the atmosphere is such that growth of the core in the nickel powder may be suppressed and coming up of a large number of nickel core may be easy. As a result, the obtained nickel powder can be prevented from forming coarse particles or agglomerating. Thus, in the present invention, after keeping at the above-described reduction temperature for the above-described time, the temperature of the reaction solution may subsequently depart from the above-described reduction temperature range. For example, to increase the rate of the reduction reaction, the temperature of the reaction solution may be allowed to exceed the above-described reduction temperature. The nickel powder according to the present invention is obtained by carrying out the above-described steps. Because the nickel powder according to the present invention is produced under the above-described conditions, the powder has the following physical properties.

Nickel powder according to the present invention:

[0031] The nickel powder according to the present invention essentially consists of nickel, and has a particle shape which is about spherical. The nickel powder according to the present invention has the average particle size obtained by an image-analysis of usually 0.02 to 0.2 micron meter, and preferably 0.03 to 0.1 micron meter. If the average particle size obtained by an image-analysis was less than 0.02 micron meter, the primary particles tend to agglomerate together, so it is not preferable. If the average particle size obtained by an image-analysis was over 0.2 micron meter, the primary particle size maximum value is too large, which makes it difficult to obtain a thin and smooth electrode film, so it is not preferable. In the present invention, an average particle size obtained by an image-analysis refers to the average particle size of 100 primary particles obtained by performing an image analysis using a high precision image analyzer IP-1000 PC manufactured by Asahi Engineering Corporation on the observed image of the sample powder at a magnification sufficient to view 100 or more primary particles in the image screen (e.g., a magnification of about 50, 000 times) using a scanning electron microscope (SEM) or a transmission electron microscope (TEM).

[0032] The nickel powder according to the present invention has an average particle size D_{50} of usually 0.1 to 0.5 micron meter, and preferably 0.2 to 0.3 micron meter. If the average particle size D_{50} was less than 0.1 micron meter, the particles are too small and the nickel powder may be easily oxidized, so it is not preferable. If the average particle size D_{50} was over 0.5 micron meter, it is difficult to make the nickel film formed from a conductive paste containing such nickel powder sufficiently thin, and the smoothness of the nickel film surface tends to be bad, so it is not preferable. In the present invention, average particle size D_{50} refers to the particle size (micron meter) at the point where the cumulative volume is 50% when it is examined by laser diffraction scattering method using a Microtrac HRA manufactured by Nikkiso Co., Ltd.

[0033] The nickel powder according to the present invention has a maximum particle size D_{max} of usually not greater than 0.7 micron meter, and preferably, not greater than 0.5 micron meter. If the maximum particle size D_{max} was over 0.7 micron meter, it is difficult to make the nickel film formed from a conductive paste containing such nickel powder sufficiently thin, and the smoothness of the nickel film surface tends to be bad, so it is not preferable. In the present invention, D_{max} refers to the maximum particle size (micronmeter) examined by laser diffraction scattering method using a Microtrac HRA manufactured by Nikkiso Co., Ltd.

[0034] Standard deviation (SD) of a particle size of the nickel powder according to the present invention is usually 0.05 to 0.2 and preferably 0.05 to 0.1. If SD of the nickel powder was within this range, it is easy to make the nickel film formed from a conductive paste containing such nickel powder sufficiently thin, and the smoothness of the nickel film surface does not easily be bad, so it is preferable. In the present invention, SD refers to the standard deviation of particle size determined when particle size distribution is examined by laser diffraction scattering method using a Microtrac HRA manufactured by Nikkiso Co., Ltd.

[0035] A carbon content of the nickel powder according to the present invention is usually 0.6% by weight or less, and preferably 0.3% by weight or less. If the carbon content was within this range, MLCC capacitance and electrode film density tend to increase as a result that the conductivity of the nickel powder increase, so it is preferable. Especially if a nickel powder was produced by the method according to the present invention as described above, the nickel powder according to the present invention has a low content or deposit of impurities, such as carbon, whereby the carbon content tends to be within the above-described range.

Conductive paste according to the present invention:

[0036] The conductive paste according to the present invention contains the above-described nickel powder according to the present invention, and in addition to the nickel powder, also contains a resin and a solvent. Examples of the resin used in the present invention may be celluloses such as ethyl cellulose, nitrocellulose and the like, and acrylic resins such as butyl methacrylate, methyl methacrylate and the like. In the present invention, the above-described resin can be used alone or in combination of two or more thereof. Further, examples of the solvent used in the present invention may be terpenes such as terpineol, dihydroterpineol and the like, and alcohols such as octanol, decanol and the like. In the present invention, the above described solvent can be used alone or in combination of two or more thereof.

[0037] The conductive paste according to the present invention has a content of the nickel powder according to the present invention of usually 40 to 70% by weight, and preferably 50 to 60% by weight. If the content of the nickel powder was within this range, the paste has good conductivity, the filling property is excellent, and shrinking by heating tends to be small, so it is preferable.

[0038] The nickel powder according to the present invention can provide a conductive paste in which the nickel powder is dispersed by mixing with a conventional paste to be used in production of the conductive paste. Such a conductive paste can be used, for example, as a nickel paste used for forming an internal electrodes of a multilayer ceramic capacitor.

[0039] While the Examples will now be shown, the present invention is not meant to be construed as being limited thereto.

[Example 1]

Premixing step:

[0040] The solution (solution A) was prepared by mixing 50 L (56 kg) of ethylene glycol (manufactured by Mitsui Chemicals Inc.), 12.47 kg of nickel hydroxide (manufactured by OM Group Inc.), 53 ml of aqueous palladium nitrate solution prepared to 100 g/l (manufactured by Tanaka Kikinzoku Group), and 0.67 kg of polyvinylpyrrolidone K30 (manufactured by Wako Pure Chemical Industries, Ltd.) and stirring in a tank.

Mixing step:

[0041] 80 L (89 kg) of ethylene glycol (manufactured by Mitsui Chemicals Inc.) was put into another tank with stirring, followed by putting the whole amount of the solution A therein and mixed to prepare the solution (solution B).

Reduction step:

[0042] The solution B was transferred to a reaction tank, followed by putting 29 L (32 kg) of ethylene glycol (manufactured by Mitsui Chemicals Inc.) with mixing to prepare the reaction solution (reaction solution A). The reaction solution A was heated and kept at 160 deg. C for 10 hours. The slurry (slurry A) was obtained as a result of these operations.

Rinsing step:

[0043] After finishing the reduction step, 140 L of the ethylene glycol in the upper portion of the slurry A was discharged from the upper portion of the reaction tank. Next, the remainder of the slurry A was filtrated with suction to carry out solid-liquid separation. The separated cake was charged with 200 L of water for decantation, and then water was separated. The powder from which water had been separated was charged with 50 L of methanol for decantation, and then methanol was separated. The nickel powder was obtained after drying the powder from which methanol had been separated for 5 hours at 80 deg. C.

[0044] The obtained nickel powder was observed with a scanning electron microscope (SEM). A scanning electron microscope photograph of the nickel powder is shown in Figure 1. Further, the average particle size obtained by an image-analysis, D_{10}, D_{50}, D_{90}, D_{max}, SD and residual carbon content of the obtained nickel powder were examined by the measurement methods described later. The results are shown in Table 1. The graph of the particle size distribution is shown in Figure 2.

Average particle size obtained by an image-analysis measurement method:

[0045] The sample powder was examined at a magnification sufficient to view 100 or more primary particles in the image screen (50, 000 times magnification) using a scanning electron microscope (SEM), followed by performing an image analysis on the obtained images using a high precision image analyzer IP-1000 PC manufactured by Asahi Engineering Corporation to obtain average particle size of 100 primary particles.

Particle size D_{10}, D_{50}, D_{90}, D_{max} and SD measurement method:

[0046] Sample solution is prepared by collecting about 0.1 g of a sample into a 200 cc sample vessel, followed by putting and mixing 100 ml of a 0.1 g/l dispersing agent (SN Dispersant 5468, manufactured by San Nopco Limited) and dispersed for 10 minutes using an ultrasonic disperser (US-300T, manufactured by Nippon Seiki Co., Ltd.). Using the sample solution, the particle size (micron meter) at the point where the cumulative volume was examined by laser diffraction scattering method using a Microtrac HRA manufactured by Nikkiso Co., Ltd. and of 10%, 50% and 90% were taken as D_{10}, D_{50} and D_{90}, and the maximum particle size was taken as D_{max}-Further, the standard deviation of the particle size distribution obtained in the examination was taken as the SD.

Residual carbon content measurement method:

[0047] Using a simultaneous carbon-sulfur analyzer EMIA-320V manufactured by Horiba Ltd. , 0.5 g of a test sample was burned by heating with an outlet setting of 175 mA, whereby the carbon content was measured from infrared adsorption.

[Comparative Example 1]

Premixing and mixing steps:

[0048] The solution B was prepared by carrying out the premixing and mixing steps in the same manner as in Example 1.

Reduction step:

[0049] The reaction solution (reaction solution A) is prepared by transferring the solution B to a reaction tank, followed by putting and mixing 29 L (32 kg) of ethylene glycol (manufactured by Mitsui Chemicals Inc.). The slurry (slurry B) was obtained after heating and keeping the reaction solution A at 190 deg. C for 5 hours.

Rinsing step:

[0050] After that, 140 L of the ethylene glycol in the upper portion of the slurry B was discharged from the upper portion of the reaction tank. Next, the remainder of the slurry B was filtrated with suction to carry out solid-liquid separation. The separated cake was charged with 200 L of water for decantation, and then water was separated. The powder from which water had been separated was charged with 50 L of methanol for decantation, and then methanol was separated. The nickel powder is obtained after the powder from which methanol had been separated was dried for 5 hours at 80 deg. C.

[0051] The obtained nickel powder was observed with a scanning electron microscope (SEM). A scanning electron microscope photograph of the nickel powder is shown in Figure 3. Further, the various properties of the obtained nickel powder were examined in the same manner as in Example 1. The results are shown in Table 1. A graph of the particle size distribution is shown in Figure 4.

[0052]

[Table 1]

	Image-analysis average particle size (micron meter)	D_{10} (micron meter)	D_{50} (micron meter)	D_{90} (micron meter)	D_{max} (micron meter)	SD	Residual carbon content (% by weight)
Ex. 1	0.092	0.17	0.27	0.41	0.69	0.09	0.5
Comp. Ex. 1	0.089	0.23	0.36	0.72	1.95	0.18	0.8

[0053] It is understood from Table 1 that the nickel powder of Example 1 has a smaller D_{max} and SD than the nickel powder of Comparative Example 1, and the residual carbon content is less also.

Industrial Applicability

[0054] The nickel powder and conductive paste according to the present invention can be used, for example, as nickel paste and a raw material thereof which can be used in the formation of internal electrodes of a multilayer ceramic capacitor.

Claims

1. A method of producing a nickel powder wherein a reaction solution comprising a nickel salt, a polyol and a noble metal catalyst is heated to a reduction temperature and nickel ion in the reaction solution are reduced while keeping the reduction temperature, **characterized in that** the reduction temperature is from 150 to 210 deg. C, and is 150 to 10 deg. C lower than a boiling point of the polyol.

2. The method of producing a nickel powder according to claim 1, **characterized in that** the reaction solution further comprises a dispersing agent.

3. A nickel powder **characterized in that** the powder is produced by the method according to claim 1 or 2.

4. The nickel powder according to claim 3, **characterized by** having average particle size obtained from an image-analysis of 0.02 to 0.2 micron meter.

5. The nickel powder according to claim 3 or 4, **characterized by** having an average particle size D_{50} of 0.1 to 0.5 micron meter.

6. The nickel powder according to any one of claims 3 to 5, **characterized by** having a maximum particle size D_{max} of not greater than 0.7 micron meter.

7. The nickel powder according to any one of claims 3 to 6, **characterized by** having a carbon content of not greater than 0.6% by weight.

8. A conductive paste **characterized by** comprising the nickel powder according to any one of claims 3 to 7.

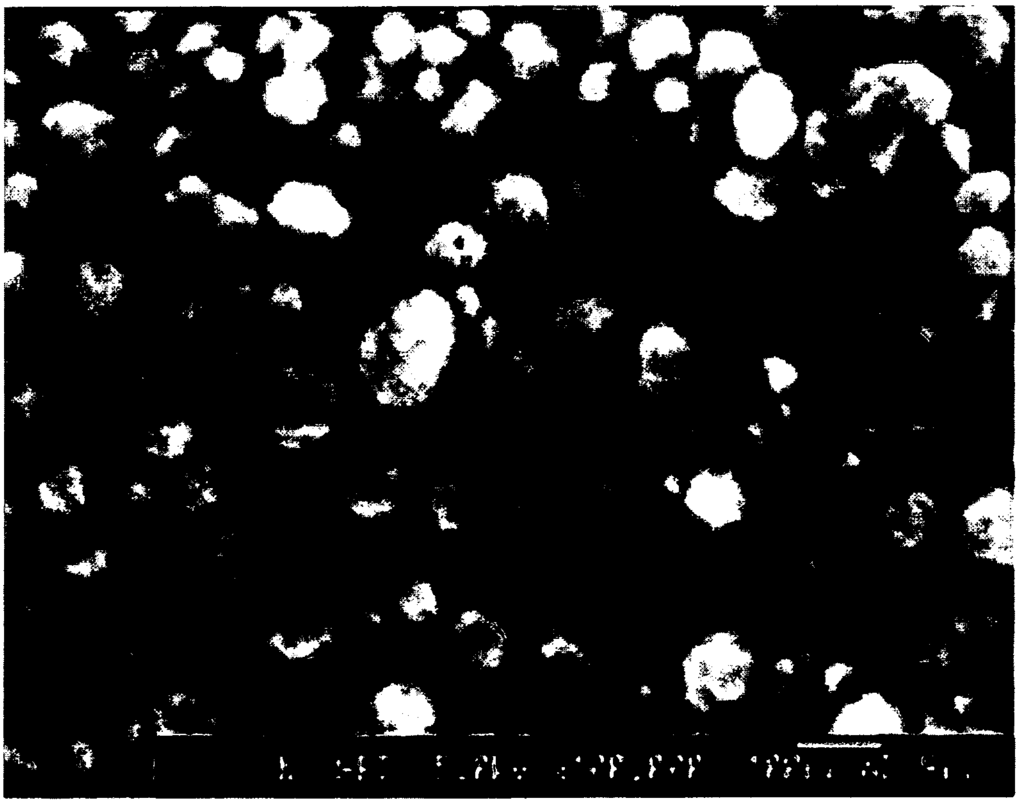
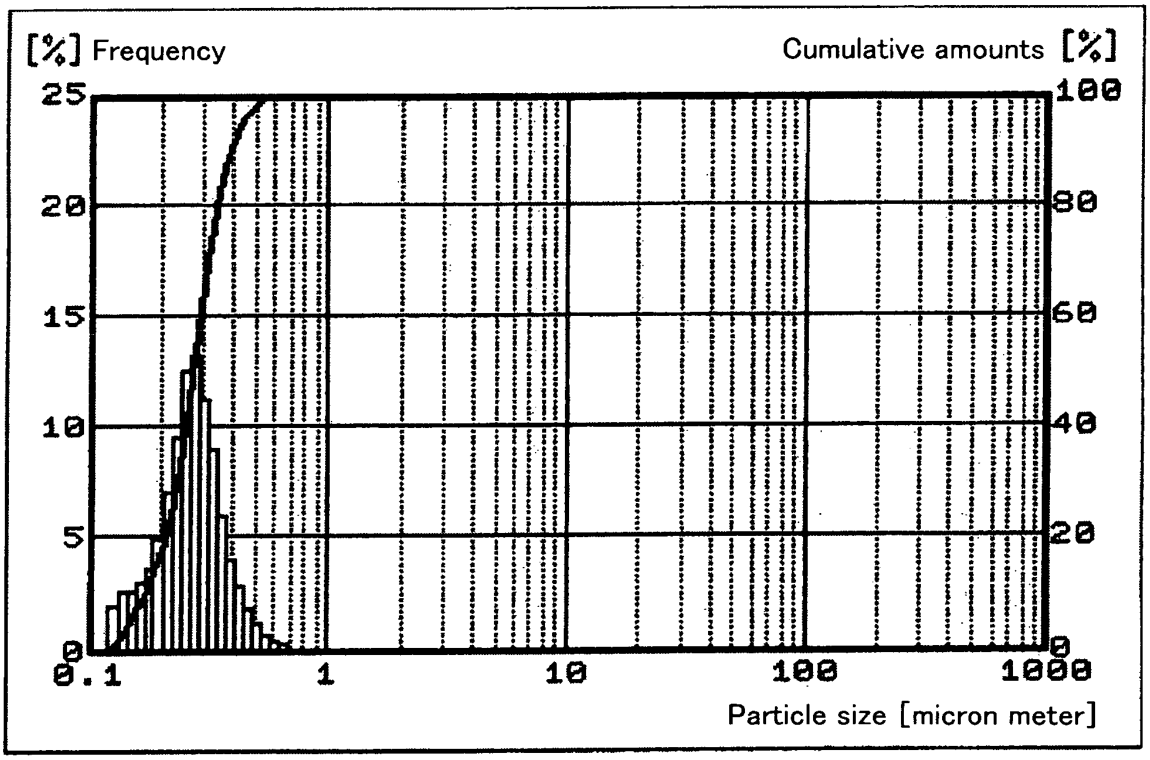


Fig. 1


[%] Frequency
Cumulative amounts [%]
25
20
15
10
5
0
100
80
60
40
20
0
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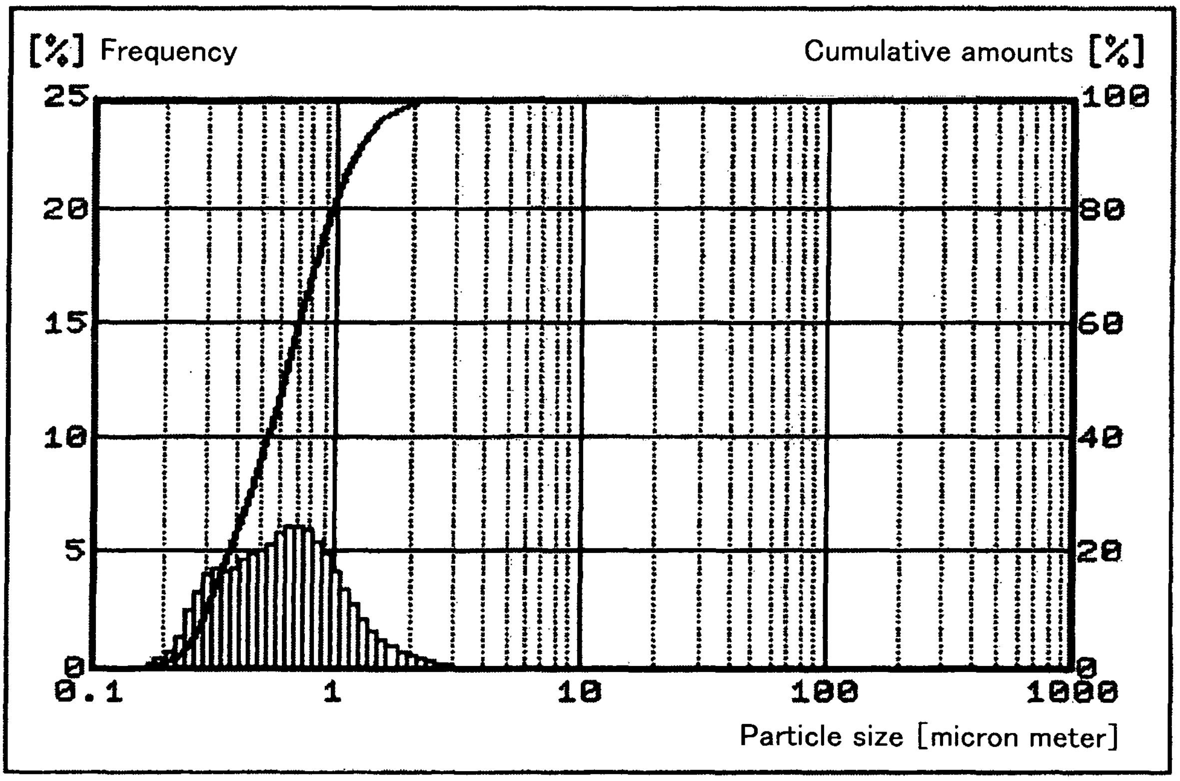
10
100
1000
Particle size [micron meter]

Fig. 2

Fig. 3


[%] Frequency
Cumulative amounts [%]
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20
15
10
5
0
100
80
60
40
20
0
0.1
1
10
100
1000
Particle size [micron meter]

Fig. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2005/022623

A. CLASSIFICATION OF SUBJECT MATTER
B22F9/24(2006.01), **B22F1/00**(2006.01), **H01B1/22**(2006.01), **H01B5/00**(2006.01)

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)
B22F1/00, B22F9/24, H01B1/22, H01B5/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2006
Kokai Jitsuyo Shinan Koho	1971-2006	Toroku Jitsuyo Shinan Koho	1994-2006

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 59-173206 A (Universite Pari VII), 01 October, 1984 (01.10.84), Claims 1 to 2; page 3, lower left column, & US 4539041 A & EP 0113281 A1	1-8
A	JP 2004-308013 A (Samsung Electronics Co., Ltd.), 04 November, 2004 (04.11.04), Claims 1, 3, 8, 10 to 12; Par. Nos. [0037] to [0038]; examples & US 2004/0200318 A1	1-8
P,A	JP 2005-154904 A (Samsung Electronics Co., Ltd.), 16 June, 2005 (16.06.05), Par. No. [0048] & US 2005/0121656 A1	1-8

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
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"&" document member of the same patent family

Date of the actual completion of the international search 02 March, 2006 (02.03.06)	Date of mailing of the international search report 14 March, 2006 (14.03.06)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (April 2005)

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- JP 59173206 A **[0004]**