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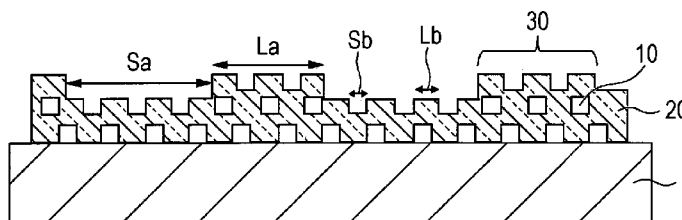
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(54) Title: OPTICAL MEMBER, IMAGE PICKUP APPARATUS, AND METHOD FOR PRODUCING OPTICAL MEMBER

FIG. 1



(57) Abstract: There is provided an optical member having a low reflectance and a good dustproof property. An optical member includes a base and a porous layer disposed on the base and including a plurality of pores. A plurality of projections are formed on a surface of the porous layer. The width of the projections is larger than the pore size of the pores of the porous layer. The distance between the projections is 75 nm or more and 300 nm or less.



DESCRIPTION

OPTICAL MEMBER, IMAGE PICKUP APPARATUS, AND METHOD FOR PRODUCING OPTICAL MEMBER

Technical Field

[0001] The present invention relates to an optical member including a porous layer on a base, an image pickup apparatus including the optical member, and a method for producing the optical member.

Background Art

[0002] In image pickup apparatuses such as digital cameras, an image pickup element such as a charge-coupled device (CCD) sensor or a complementary metal oxide semiconductor (CMOS) sensor receives imaging light beams and outputs photoelectrically converted signals. The signals are converted into image data, and the data is stored in a recording medium such as a memory card. In such image pickup apparatuses, an optical filter such as a low-pass filter or an infrared cut filter is disposed on the object side of the image pickup element.

[0003] In particular, in lens-replaceable digital cameras, mechanically operating parts such as a shutter are disposed near an optical filter, and foreign matter such as dust generated from the operating parts may adhere to the optical

filter. When a lens is replaced, dust and the like present outside the digital camera may enter the main body of the digital camera through an aperture of a lens mount and adhere to the optical filter. If dust adheres to the optical filter, portions to which the dust adheres are taken in an image as black spots, which may degrade the quality of the image.

[0004] PTL 1 discloses that a foreign matter adhesion-preventing film composed of a material containing fluorine is formed on the surface of an optical filter in order to suppress the adhesion of dust. PTL 2 discloses that a dustproof film having a fine uneven structure constituted by a petaloid alumina film is formed on a light transmissive member.

Citation List

Patent Literature

[0005] PTL 1 Japanese Patent Laid-Open No. 2006-163275

PTL 2 Japanese Patent Laid-Open No. 2007-183366

Summary of Invention

Technical Problem

[0006] The foreign matter adhesion-preventing film described in PTL 1 improves the dustproof property, but increases the reflectance at the surface. The dustproof film described in PTL 2 may degrade the reflectivity and the dustproof property because the dustproof film has low

mechanical strength and thus the uneven structure is easily broken.

[0007] The present invention provides an optical member having a low reflectance and a good dustproof property, an image pickup apparatus, and a method for producing the optical member.

Solution to Problem

[0008] An optical member according to an aspect of the present invention includes a base and a porous layer disposed on the base and including a plurality of pores. A plurality of projections are formed on a surface of the porous layer. The width of the projections is larger than the pore size of the pores of the porous layer. The distance between the projections is 75 nm or more and 300 nm or less.

Advantageous Effects of Invention

[0009] According to the present invention, there can be provided an optical member having a low reflectance and a good dustproof property, an image pickup apparatus, and a method for producing an optical member.

Brief Description of Drawings

[0010] Fig. 1 schematically shows an example of an optical member according to an embodiment of the present invention.

[0011] Figs. 2A and 2B are diagrams for describing liquid bridge.

[0012] Fig. 3 is a diagram for describing porosity.

[0013] Figs. 4A and 4B are diagrams for describing the pore size and skeleton size.

[0014] Fig. 5 schematically shows an example of an image pickup apparatus including the optical member according to an embodiment of the present invention.

[0015] Figs. 6A to 6D schematically show an example of a method for producing an optical member according to a first embodiment.

[0016] Figs. 7A to 7D schematically show an example of a method for producing an optical member according to a second embodiment.

[0017] Fig. 8 shows a surface profile of an optical member 1 produced in Example 1.

[0018] Fig. 9 shows a surface profile of an optical member 4 produced in Comparative Example 1.

[0019] Fig. 10 shows the wavelength dependence of the reflectances of optical members in Examples 1 to 3 and Comparative Example 1 and a base.

Description of Embodiments

[0020] The present invention will now be described in detail on the basis of embodiments of the present invention. Well-known or publicly known techniques in the technical field concerned are applied to components that are not particularly illustrated or described in this specification.

Optical member

[0021] As shown in Fig. 1, an optical member according to an embodiment of the present invention includes a base 1 and a porous layer 20 that is disposed on the base 1 and includes a plurality of pores 10. A plurality of projections 30 are formed on a surface of the porous layer 20. The width La of the projections 30 is larger than the pore size Sb of the pores 10. The distance Sa between the plurality of projections 30 is 75 nm or more and 300 nm or less.

[0022] Since the porous layer 20 includes the plurality of pores 10, the refractive index of the porous layer 20 is lower than that of the base 1. Therefore, the reflectance is reduced compared with a structure including no porous layer 20 because the reflection at the surface (porous layer 20 side) of the optical member is suppressed.

[0023] Since the width La of the projections 30 on the surface of the porous layer 20 is larger than the pore size Sb of the pores 10 and the distance Sa between the plurality of projections 30 is 75 nm or more and 300 nm or less, an adhesive force exerted on dust is low and a good dustproof property can be achieved.

[0024] The inventors of the present invention assume the mechanism of improving the dustproof property to be as follows. In general, when no irregularities are formed on

the surface, a force (adhesive force) that attracts dust is generated on the entire surface. On the other hand, in the porous layer 20 according to an embodiment of the present invention, such an adhesive force is generated only on the projections 30 of the porous layer 20.

[0025] Figs. 2A and 2B schematically show the adhesive force generated by liquid bridge. If a liquid 73 is present between an object 71 (or 81) and a dust 72, liquid bridge is formed between the object 71 (or 81) and the dust 72. The pressure is different between the inside (liquid side) and the outside (air side) of the air interface of the liquid bridge, and the pressure on the liquid side is lower than that on the air side. The pressure on the air side is equivalent to the atmospheric pressure and the pressure on the liquid side is a negative pressure, which is lower than the atmospheric pressure. The negative pressure P is represented by formula 1. The adhesive force F is a value obtained by multiplying the negative pressure P by the contact area S with the dust 72, which is represented by formula 2. Herein, R_1 represents a radius of curvature of the air interface of the liquid 73 formed between the object 71 (or 81) and the dust 72. R_2 represents a radius of a contact region between the object 71 (or 81) and the liquid 73. The contact area S is represented by a product of a surface area S_0 of the object 71 (or 81) and a ratio β of the

projections on the surface. Furthermore, σ is a constant.

$$P = \sigma(1/R_1 - 1/R_2) \cdots \text{Formula 1}$$

$$F = PS = PS_0\beta \cdots \text{Formula 2}$$

[0026] Fig. 2A shows the case where the surface of the object 71 is a smooth surface. In Fig. 2A, R_2 corresponds to a radius R of the dust 72. Fig. 2B shows the case where the object 81 has a plurality of projections on its surface. In Fig. 2B, R_2 corresponds to a half width R' of a projection. The projection corresponds to each of the projections 30 in Fig. 1.

[0027] As is clear from the formula 1, the adhesive force is decreased by bringing R_2 close to R_1 , that is, by decreasing the contact area between the object 71 (or 81) and the liquid 73.

[0028] Therefore, the width La of the projections 30 of the porous layer 20 may be decreased. However, if the width La of the projections 30 is smaller than or equal to the pore size Sb of the pores 10, in particular, smaller than 5 nm, the mechanical strength of the optical member decreases and the projections 30 are easily broken, which makes it difficult to maintain a low reflectance and a good dustproof property. If the width La of the projections 30 is larger than 500 nm, the contact area between the projections 30 and the dust 72 increases, which makes it difficult to achieve an effect of reducing an adhesive force by liquid bridge.

Therefore, the width L_a of the projections 30 is preferably 5 nm or more and 500 nm or less and more preferably 5 nm or more and 300 nm or less.

[0029] As shown by the formula 2, the adhesive force can also be decreased by decreasing the ratio β of the projections on the surface. A small ratio β can be achieved not only by decreasing the width L_a of the projections 30 but also by increasing the distance S_a between the projections 30. From this point of view, the distance S_a between the projections 30 can be 75 nm or more. Herein, if the distance S_a between the projections 30 is more than 300 nm, droplets enter the spaces between the projections 30 and the liquid is not divided between the projections 30. Consequently, an effect of scattering liquid bridge by the projections 30 is not easily achieved. Therefore, the distance S_a between the projections 30 can be 75 nm or more and 300 nm or less.

[0030] The width L_a of the projections 30 and the distance S_a between the projections 30 can be measured by the following method. First, the surface profile of the optical member is measured with an atomic force microscope (hereafter abbreviated as "AFM") (E-Sweep manufactured by Seiko Instruments Inc.). A cross-sectional image in a scanning direction (horizontal direction) of the AFM is obtained from the measurement data of the surface profile.

In the obtained image, projections having a height of 5 nm or more are defined as the projections 30. The reason why projections having a height of 5 nm or more are defined as the projections 30 is that, if the height of the projections 30 is less than 5 nm, an effect of scattering liquid bridge is not achieved and thus a good dustproof property is not achieved. At least 20 widths of the projections 30 at a position 5 nm from the peaks of the projections 30 are measured in the cross-sectional image. The average of the measured widths is defined as the width L_a of the projections 30. Furthermore, at least 20 distances between the peaks of the projections 30 are measured. The average of the measured distances is defined as the distance S_a .

[0031] The ratio L_a/S_a of the width of the projections 30 to the distance between the projections 30 is preferably less than 1.00 and more preferably less than 0.60. When the ratio L_a/S_a is less than 1.00, the contact area between the surface of the optical member and an adherent can be sufficiently reduced.

[0032] The pores 10 of the porous layer 20 can be present on the surface (the surface opposite the base 1) of the porous layer 20. The exposure of the pores 10 at the surface produces combined effects of suppressing liquid bridge by the projections 30 and suppressing liquid bridge by the pores 10. Thus, a good dustproof property can be

achieved. In this case, portions other than the pores 10 in Fig. 1 also correspond to the projections in Fig. 2B.

[0033] The porous layer 20 may be composed of any known porous material as long as the porous layer 20 is within the scope of the present invention. The porous layer 20 can be, for example, a porous glass layer that uses a phase separation phenomenon, a porous silica layer such as mesoporous silica, or a porous polymer layer. In particular, a porous glass layer that uses spinodal phase separation and has a structure in which pores are three-dimensionally connected can be suitably used as the porous layer 20 because such a porous glass layer has both high reflectance and high strength.

[0034] The "phase separation" will be described on the basis of the case of borosilicate glass containing silicon oxide, boron oxide, and an alkali metal-containing oxide in a glass body. The "phase separation" means that the composition in glass before phase separation is separated into a phase (non-silicon-oxide-rich phase) containing an alkali metal-containing oxide and boron oxide in amounts larger than those in the composition before phase separation and a phase (silicon-oxide-rich phase) containing an alkali metal-containing oxide and boron oxide in amounts smaller than those in the composition before phase separation. The glass subjected to the phase separation is etched to remove

the non-silicon-oxide-rich phase. Thus, a porous structure is formed in the glass body.

[0035] Phase separation is classified into spinodal phase separation and binodal phase separation. The pores of the porous glass obtained by spinodal phase separation are through-pores that extend from the surface to the inside. More specifically, the porous structure derived from spinodal phase separation is an "ant's nest" structure in which pores are three-dimensionally interconnected. The skeleton formed of silicon oxide corresponds to "walls" and the through-pores correspond to "cavities".

[0036] The porous glass obtained by binodal phase separation has a structure in which independent pores each enclosed by a closed surface with a substantially spherical shape are discontinuously present in the skeleton formed of silicon oxide.

[0037] The pores derived from spinodal phase separation and the pores derived from binodal phase separation can be differentiated by morphological observation with an electron microscope. Occurrence of spinodal phase separation or binodal phase separation is determined by the composition of the glass body and the phase separation temperature.

[0038] A porous structure derived from spinodal phase separation has a three-dimensional network of through-pores extending from the surface to the inside and has porosity

that can be controlled by changing the heat-treatment conditions. The porous structure has a three-dimensional intricate winding skeleton and a plurality of pores interconnected in three dimensions around the three-dimensional skeleton. The porous structure can therefore have high strength even at high porosity. Since the porous structure can have high surface strength even at high porosity, it is possible to provide an optical member that has excellent antireflection performance and high scratch resistance.

[0039] The porosity of the porous layer 20 is preferably 20% or more and 70% or less and more preferably 20% or more and 60% or less. If the porosity is less than 20%, the advantages of the porous layer 20 cannot be sufficiently utilized. If the porosity is more than 70%, the surface strength unfavorably tends to decrease. The porosity of the porous layer 20 in the range of 20% or more and 70% or less corresponds to a refractive index of 1.10 or more and 1.40 or less.

[0040] The porosity can be measured by the following measurement method. An electron micrograph image is binarized with respect to a skeleton and pores. Specifically, the surface of the porous layer 20 is observed with a scanning electron microscope (FE-SEM S-4800, manufactured by Hitachi, Ltd.) at an accelerating voltage of

5.0 kV at a magnification of 100,000 times (or 50,000 times) at which it is easy to observe the skeleton on a gray scale. The observed SEM image is stored and converted into a graph showing the frequency as a function of image density with image analysis software. Fig. 3 is a graph showing the frequency as a function of image density in the porous layer 20. The image density at the peak indicated by a down arrow in Fig. 3 corresponds to the skeleton on the front surface. A bright portion (skeleton) and a dark portion (pores) are binarized into black and white using an inflection point close to the peak as a threshold. The ratio of a black area to the entire area (the total of white and black areas) is determined for each of the black areas in the image. The ratios are averaged to determine porosity.

[0041] The pore size S_b of the porous layer 20 is preferably 5 nm or more and 50 nm or less and more preferably 5 nm or more and 20 nm or less. If the pore size S_b is less than 5 nm, the features of the structure of the porous layer 20 cannot be sufficiently utilized. If the pore size S_b is more than 50 nm, the surface strength unfavorably tends to decrease. The pore size S_b is more preferably 20 nm or less because light scattering is considerably suppressed. Furthermore, the pore size S_b can be smaller than the thickness of the porous layer 20.

[0042] The pore size S_b is defined as an average of minor

axis lengths of ellipses corresponding to pores in a region of $5\ \mu\text{m} \times 5\ \mu\text{m}$ of any cross section of the porous layer 20. Specifically, as shown in Fig. 4A, the pore size S_b is determined by calculating the average of minor axis lengths 12 of ellipses 11 corresponding to pores 10 using an electron micrograph of a surface of a porous glass layer formed as a result of spinodal phase separation. The average is determined from at least 30 measurements.

[0043] The skeleton size L_b of the porous layer 20 is preferably 5 nm or more and 50 nm or less and more preferably 5 nm or more and 20 nm or less. If the skeleton size L_b is more than 50 nm, light scattering markedly occurs, which considerably decreases transmittance. If the skeleton size L_b is less than 5 nm, the strength of the porous layer 20 tends to decrease. Furthermore, the skeleton size L_b can be 20 nm or less because light scattering is suppressed. When the skeleton size L_b is within the above range, sufficient strength is achieved even at high porosity unlike a petaloid structure. Therefore, an optical member that stably exhibits a low reflectance and a good dustproof property can be provided.

[0044] The skeleton size L_b is defined as an average of minor axis lengths of ellipses corresponding to skeleton walls in a region of $5\ \mu\text{m} \times 5\ \mu\text{m}$ of any cross section of the porous layer 20. Specifically, as shown in Fig. 4B, the

skeleton size L_b is determined by calculating the average of minor axis lengths 15 of ellipses 14 corresponding to skeleton walls 13 using an electron micrograph of a surface of the porous layer 20 formed as a result of spinodal phase separation. The average is determined from at least 30 measurements.

[0045] It should be noted that light scattering is affected by various factors such as the thickness of an optical member and does not uniquely depend on the pore size S_b and the skeleton size L_b .

[0046] The thickness of the porous layer 20 is not particularly limited, but is preferably 0.1 μm or more and 20.0 μm or less and more preferably 0.1 μm or more and 10.0 μm or less.

[0047] If the thickness is less than 0.1 μm , an effect produced by high porosity (low refractive index) is not achieved. If the thickness is more than 20.0 μm , the influence of scattering increases, which makes it difficult to use the porous layer 20 for an optical member.

[0048] The thickness of the porous layer 20 is determined by the following method. Specifically, a SEM image (electron micrograph) is taken with a scanning electron microscope (FE-SEMS-4800, manufactured by Hitachi, Ltd.) at an accelerating voltage of 5.0 kV. The thickness of the porous layer 20 on the base 1 is measured at 30 or more

points on the image. The average of the thicknesses is used as the thickness of the porous layer 20.

[0049] The porous layer 20 may have a monolayer structure or a multi-layer structure as long as the surface (the surface opposite the base 1) of the porous layer 20 includes the plurality of projections 30 as described above. In the entire porous layer 20, the porosity can increase in a direction from the base 1 toward the surface of the porous layer 20 to further achieve an effect of low reflectance.

[0050] In addition to the members and structures described above, the optical member according to an embodiment of the present invention may further include layers for imparting various functions. For example, a water-repellent layer composed of a fluoroalkylsilane, an alkylsilane, or the like can be disposed to impart water repellency. A dustproof property that has not been achieved can be realized by employing the structure of the present invention. By controlling the surface properties of projections, a better dustproof property can be realized due to a combined effect of the structure and the surface properties.

[0051] The base 1 and the porous layer 20 are not necessarily in contact with each other, and an intermediate layer may be formed between the base 1 and the porous layer 20. The intermediate layer desirably has a refractive index between refractive indices of the base 1 and the porous

layer 20. The intermediate layer may have a monolayer structure or a multi-layer structure in which the refractive index decreases in a direction from the base 1 toward the porous layer 20. The intermediate layer may be a non-porous layer.

[0052] The base 1 can be composed of any material suitable for the purpose. The base 1 can be composed of, for example, quartz glass or rock crystal in terms of transparency, heat resistance, and strength. The base 1 may have a layered structure composed of different materials. The base 1 may also be composed of a material for low-pass filters, infrared cut filters, and lenses. The base 1 is composed of a non-porous material.

[0053] The base 1 can be transparent. The transmittance of the base 1 is preferably 50% or more and more preferably 60% or more in a visible region (wavelength range of 450 nm or more and 650 nm or less). If the transmittance is less than 50%, some problems may be posed when the base 1 is used for an optical member. The haze of the base 1 can be 0.2% or less.

[0054] The optical member according to an embodiment of the present invention may be optical members used in various displays for television sets and computers, polarizing plates for liquid crystal displays, viewing lenses for cameras, prisms, fly-eye lenses, and toric lenses. The

optical member according to an embodiment of the present invention may also be various lenses used in image-taking optical systems using the above optical members, optical systems for observation, such as binoculars, projection optical systems for liquid crystal projectors, and scanning optical systems for laser-beam printers.

[0055] The optical member according to an embodiment of the present invention may also be used in image pickup apparatuses such as digital cameras and digital video cameras. Fig. 5 is a schematic sectional view showing a camera (image pickup apparatus) including the optical member according to an embodiment of the present invention, more specifically, an image pickup apparatus configured to form an object image on an image pickup element through a lens and an optical filter.

[0056] An image pickup apparatus 300 includes a main body 310 and a detachable lens 320. An image pickup apparatus, such as a digital single-lens reflex camera, can take images at various view angles by replacing an image-taking lens to other image-taking lenses having different focal lengths. The main body 310 includes an image pickup element 311, an infrared cut filter 312, a low-pass filter 313, and an optical member 203 according to an embodiment of the present invention. The optical member 203 includes the base 1 and the porous layer 20 as shown in Fig. 1.

[0057] The optical member 203 and the low-pass filter 313 may be integrally disposed or separately disposed. The optical member 203 may also serve as a low-pass filter. That is, the base 1 of the optical member 203 may be a low-pass filter.

[0058] The image pickup element 311 is accommodated in a package (not shown) while being hermetically sealed with a cover glass (not shown). The space between the optical filters, such as the low-pass filter 313 and the infrared cut filter 312, and the cover glass is hermetically sealed with a sealing member such as a double-sided tape (not shown). Although the case where the optical filter includes both the low-pass filter 313 and the infrared cut filter 312 is described, the optical filter may be one of the low-pass filter 313 and the infrared cut filter 312.

[0059] Since the optical member 203 according to an embodiment of the present invention has an uneven structure near its surface, the optical member 203 is highly dustproof, for example, it is capable of preventing dust adhesion.

[0060] Thus, the optical member 203 is disposed on the optical filter so as to be located on the side opposite to the image pickup element 311. The optical member 203 is disposed so that the porous layer 20 is farther from the image pickup element 311 than the base 1. In other words, the optical member 203 can be disposed so that the base 1

and the porous layer 20 are located in that order from the image pickup element 311 side. The optical member 203 and the image pickup element 311 are disposed so that an image that has passed through the optical member 203 can be taken by the image pickup element 311.

[0061] The image pickup apparatus 300 according to an embodiment of the present invention may include a dust-removing device (not shown) for removing dust by generating vibration or the like. The dust-removing device includes, for example, a vibrating member and a piezoelectric element.

[0062] The dust-removing device may be disposed at any position between the image pickup element 311 and the optical member 203. For example, the vibrating member may be disposed so as to be in contact with the optical member 203, the low-pass filter 313, or the infrared cut filter 312. In particular, when the vibrating member is disposed so as to be in contact with the optical member 203, dust can be more efficiently removed because dust does not easily adhere to the optical member 203 according to an embodiment of the present invention.

[0063] The vibrating member of the dust-removing device may be provided integrally with an optical filter such as the optical member 203, the low-pass filter 313, or the infrared cut filter 312. The vibrating member may be constituted by the optical member 203 or may have a function

of the low-pass filter 313, the infrared cut filter 312, or the like.

Method for producing optical member

[0064] The optical member according to an embodiment of the present invention may be produced by any method as long as the optical member that satisfies the scope of the present invention can be produced. The production method according to an embodiment of the present invention will be described below, but is not limited thereto.

[0065] The method for producing an optical member according to an embodiment of the present invention includes a step of forming a porous layer including a plurality of pores on a base and a step of forming a plurality of projections on a surface of the porous layer. The projections are formed so that the width of the projections is larger than the pore size of the pores of the porous layer and the distance between the plurality of projections is 75 nm or more and 300 nm or less. The step of forming a plurality of projections on a surface of the porous layer may be performed after the step of forming a porous layer including a plurality of pores on a base or may be simultaneously performed with part of the step of forming a porous layer including a plurality of pores on a base.

First Embodiment

[0066] In this embodiment, an example of the method for

producing an optical member will be described below based on the case where the porous layer is a porous glass layer formed as a result of phase separation. In this case, the step of forming a porous layer includes the following steps:

- (1) a step of forming a glass powder layer 21 containing a plurality of glass powder particles on a base 1;
- (2) a step of forming a mother glass layer 22 by fusing the plurality of glass powder particles of the glass powder layer 21;
- (3) a step of forming a phase-separated glass layer 23 by subjecting the mother glass layer 22 to phase separation; and
- (4) a step of forming a porous glass layer 200 by etching the phase-separated glass layer 23.

The step of forming a plurality of projections on a surface of the porous layer (porous glass layer 200) may be performed after the step (4) or may be simultaneously performed with the step (4). The production method will be described in detail with reference to Figs. 6A to 6D.

(1) Step of forming glass powder layer

[0067] As shown in Fig. 6A, a glass powder layer 21 containing a plurality of glass powder particles is formed on a base 1. The composition of the glass powder particles may be suitably set in accordance with the optical member.

[0068] The glass powder layer 21 is formed by any method that enables film formation, such as a printing method, a spin coating method, or a dip coating method. Among them, a printing method that uses screen printing is suitably used as a method for forming a glass powder layer 21 having a desired glass composition.

[0069] Hereafter, a description will be made on the basis of a typical method that uses screen printing. In screen printing, glass powder particles in the form of a paste are prepared and printed with a screen printing machine. Therefore, a paste of the glass powder particles needs to be prepared.

[0070] A base glass to be formed into glass powder particles can be produced by a publicly known method. For example, the base glass can be produced by heat-melting raw materials containing component sources and, if necessary, shaping the molten product into a desired form.

[0071] Any glass powder particles may be used as long as they are phase-separable glass powder particles.

[0072] The heating temperature during the heat melting may be suitably set in accordance with the composition of the raw materials and the like, but is preferably 1350°C or higher and 1450°C or lower and particularly preferably 1380°C or higher and 1430°C or lower.

[0073] In order to use the base glass in the form of a

paste, the base glass is converted into glass powder particles. The glass powder particles may be produced by any publicly known method. Examples of the method include liquid-phase pulverization using a bead mill and gas-phase pulverization using a jet mill. In addition to the glass powder particles, the paste contains a thermoplastic resin, a plasticizer, and a solvent.

[0074] The content of the glass powder particles in the paste is preferably 30.0% by weight or more and 90.0% by weight or less and more preferably 35.0% by weight or more and 70.0% by weight or less.

[0075] The thermoplastic resin contained in the paste can increase the film strength after drying and impart flexibility to the film. Examples of the thermoplastic resin include polybutyl methacrylate, polyvinyl butyral, polymethyl methacrylate, polyethyl methacrylate, and ethylcellulose. These thermoplastic resins may be used alone or in combination of two or more.

[0076] Examples of the plasticizer contained in the paste include butyl benzyl phthalate, dioctyl phthalate, diisooctyl phthalate, dicapryl phthalate, and dibutyl phthalate. These plasticizers may be used alone or in combination of two or more.

[0077] Examples of the solvent contained in the paste include terpeneol, diethylene glycol monobutyl ether acetate,

and 2,2,4-trimethyl-1,3-pentanediol monoisobutyrate. These solvents may be used alone or in combination of two or more.

[0078] The paste can be prepared by kneading these materials at a predetermined ratio. The thus-prepared paste is applied onto a base by screen printing to form a glass powder layer. Specifically, the paste is applied and is then dried to remove the solvent in the paste, thereby forming a glass powder layer 21.

[0079] The temperature and time for removing the solvent by drying can be suitably changed in accordance with the type of solvent. However, it is desirable to dry the paste at a temperature lower than the decomposition temperature of the thermoplastic resin. If the drying temperature is higher than the decomposition temperature of the thermoplastic resin, the glass particles are not fixed and the glass powder layer 21 tends to have defects.

[0080] Use of the base 1 suppresses the strain of the glass layer caused by heat treatment in the phase separation step and facilitates the thickness control of the porous glass layer 200.

[0081] The softening temperature of the base 1 is preferably higher than or equal to the heating temperature in the phase separation step described below (phase separation temperature) and more preferably higher than or equal to the phase separation temperature + 100°C. In the

case where the base 1 is made of crystals, however, the softening temperature of the base 1 is the melting temperature. When the softening temperature is lower than the phase separation temperature, the base 1 may be unfavorably distorted in the phase separation step.

[0082] The base 1 can have resistance to etching of the phase-separated glass layer 23 described below. For example, the base 1 can be composed of quartz glass or rock crystal.

(2) Step of forming mother glass layer

[0083] As shown in Fig. 6B, the glass powder layer 21 is then heated to fuse the glass powder particles, thereby forming a phase-separable mother glass layer 22 on the base 1. The term "phase-separable" means that the phase separation described above can occur at a certain heating temperature.

[0084] The glass powder particles can be fused by performing a heat treatment at a temperature higher than or equal to the glass transition temperature T_g (°C). In the phase-separable glass powder particles, a heat treatment is performed at a temperature higher than or equal to the crystallization temperature T_c (°C), whereby voids in the film are reduced and a uniform film is formed.

[0085] The fusing temperature is determined in accordance with the type of glass and thus does not limit the present invention. The fusing temperature suitably set in typical

phase-separable glasses is 600°C or higher and 1200°C or lower. However, in the present invention, the fusing temperature is higher than or equal to the crystallization temperature and 1200°C or lower in order to suppress the formation of voids. If the fusing temperature is higher than 1200°C, the composition of glass changes and phase separation sometimes does not occur.

[0086] The heating time required for fusing the glass powder particles can be suitably set in accordance with the heating temperature, but can be 5 minutes or longer and 50 hours or shorter.

[0087] The heating rate until the fusing temperature can be suitably set in accordance with the mother glass layer 22.

[0088] A publicly known heat treatment method can be used as a heating method for fusion. The heat treatment method may involve the use of an electric furnace, an oven, or infrared radiation. Any heating methods such as convective, radiant, and electric heating methods can be employed. In particular, an infrared radiation furnace is suitably used in terms of facilitation of fusion of the glass powder particles.

[0089] When the firing atmosphere is an oxygen-rich atmosphere (an oxygen concentration of 50% or more), a binder resin component is effectively decomposed, and thus voids resulting from the binder resin component in the film

can be reduced. The solvent in the paste may be removed simultaneously in the fusion of the glass powder layer.

[0090] After the mother glass layer 22 is formed, a surface of the mother glass layer 22 may be flattened. Specifically, it is desirable to polish a surface of the mother glass layer 22. The flattening may be performed after a phase-separated glass layer described below is formed. Surface flattening may be performed only after the mother glass layer 22 is formed or only after the phase-separated glass layer is formed, or both after the mother glass layer 22 is formed and after the phase-separated glass layer is formed.

(3) Step of forming phase-separated glass layer

[0091] As shown in Fig. 6C, the mother glass layer 22 is then subjected to phase separation to form a phase-separated glass layer 23 on the base 1.

[0092] More specifically, the phase separation step of forming a phase-separated glass layer 23 is performed at a temperature of 450°C or higher and 750°C or lower for 3 hours or longer and 100 hours or shorter. The heating temperature in the phase separation step is not necessarily fixed, and may be continuously changed or may include different temperature stages.

[0093] The porosity of the porous glass layer 200 described below can be adjusted by controlling the phase

separation treatment time.

[0094] Since optical members require a very low haze, the porous glass layer 200 for use in optical members can have a small skeleton size and a very fine structure of pores to decrease the haze.

[0095] Heating in the phase separation treatment may be performed by a publicly known heat treatment method. The heat treatment method may involve the use of an electric furnace, an oven, or infrared radiation. Any heating methods such as convective, radiant, and electric heating methods can be employed.

(4) Step of forming porous glass layer

[0096] As shown in Fig. 6D, the phase-separated glass layer 23 is then etched to form the porous glass layer 200 on the base 1.

[0097] The non-silicon-oxide-rich phase in the phase-separated glass layer can be removed by an etching treatment while a silicon-oxide-rich phase is left. The silicon-oxide-rich phase forms a skeleton of the porous glass layer, and portions from which the non-silicon-oxide-rich phase has been removed form pores of the porous glass layer 200.

[0098] The etching treatment for removing the non-silicon-oxide-rich phase is generally a treatment (wet etching) in which a soluble non-silicon-oxide-rich phase is eluted by bringing it into contact with an aqueous solution. A glass

is generally brought into contact with the aqueous solution by immersing the glass in the aqueous solution. However, any method for bringing a glass into contact with an aqueous solution can be employed. For example, an aqueous solution is applied to a glass. An aqueous solution required for the etching treatment may be a known solution that can elute the non-silicon-oxide-rich phase, such as water, an acid solution, or an alkaline solution. For some applications, a plurality of processes of bringing a glass into contact with an aqueous solution may be selected.

[0099] The aqueous solution can be a solution of an acid, for example, an inorganic acid such as hydrochloric acid or nitric acid. The acid solution can be normally an aqueous solution containing water as a solvent. The concentration of the acid solution may be normally in the range of 0.1 mol/L or more and 2.0 mol/L or less. In an acid treatment process using the acid solution, the acid solution temperature may be in the range of 15°C or higher and 100°C or lower, and the processing time may be in the range of 1 hour or longer and 500 hours or shorter.

[0100] Depending on the glass composition and the production conditions, a silicon oxide layer having a thickness of 20 nm to 30 nm may be formed on a glass surface after the phase separation treatment. The silicon oxide layer inhibits etching. The silicon oxide layer on the

surface can be removed by polishing or an acid or alkaline treatment.

[0101] In particular, polishing can be employed because the flatness of a surface of an optical member is achieved and the haze (scattering) can be reduced.

[0102] The treatment with an acid solution or an alkaline solution can be followed by a water treatment. A water treatment can suppress the deposition of residual components onto the skeleton of the porous glass layer 200 and tends to increase the porosity of the porous layer and suppress scattering.

[0103] The temperature in the water treatment can generally be in the range of 15°C or higher and 100°C or lower. The time for the water treatment can be suitably determined in accordance with, for example, the composition and size of the glass to be treated and may be in the range of 1 hour or longer and 50 hours or shorter.

Step of forming a plurality of projections on surface of porous layer

[0104] Finally, a plurality of projections 30 are formed on a surface of the porous glass layer 200. This step may be performed simultaneously with the wet etching conducted in the step of forming a porous glass layer 200 or may be performed by any known method such as dry etching or mechanical polishing after the step of forming a porous

glass layer 200. In particular, overetching can be performed in the step of forming a porous glass layer 200. The term "overetching" means that part of the skeleton is excessively dissolved by wet etching. As a result of this overetching, an uneven structure (projections) is formed on the surface of the porous glass layer 200.

[0105] In the production method according to this embodiment, since the glass powder particles are fused, a composition difference due to the grain boundaries between the glass powder particles and an uneven structure derived from the glass powder particles are believed to tend to remain on the surface of the phase-separated glass layer 23. When such a phase-separated glass layer 23 is subjected to overetching, the degree of overetching varies due to the composition difference and the uneven structure, and thus a plurality of projections 30 can be easily formed on the surface of the porous glass layer 200. Furthermore, as a result of the overetching, part of the skeleton is dissolved, which decreases the density of the skeleton of the porous glass layer 200. This increases the porosity and thus decreases the refractive index of the porous glass layer 200. Consequently, a porous glass layer 200 having a lower refractive index than typical porous glass layers can be realized.

[0106] In order to form the projections 30 so that the

width of the projections 30 is larger than the pore size of the pores of the porous glass layer 200 and the distance between the plurality of projections 30 is 75 nm or more and 300 nm or less, the overetching is performed under the following conditions. That is, in the step of forming a porous glass layer 200 described above, the etching temperature may be increased or the etching time may be lengthened. Specifically, in the acid treatment process that uses an acid solution, the acid solution temperature may be in the range of 80°C or higher and 100°C or lower or the treatment time may be in the range of 20 hours or longer and 500 hours or shorter. In particular, the etching temperature can be increased because a high etching rate is achieved and the projections 30 are easily formed on the surface of the porous glass layer 200 in accordance with the surface profile of the phase-separated glass layer 23.

Second Embodiment

[0107] In addition to the method described in the first embodiment, the following method can be employed as the method for forming a plurality of projections 30 on a surface of the porous glass layer 200. That is, in the above-described step (2), a plurality of projections 31 are formed on the surface of the mother glass layer, and the steps (3) and (4) are performed while the projections 31 on the surface are retained. In other words, the method for

producing an optical member according to this embodiment includes the following steps. Figs. 7A to 7D show an example of the production method according to the second embodiment.

(1) a step of forming a glass powder layer 21 containing a plurality of glass powder particles on a base 1

(2A) a step of forming a mother glass layer 42 by fusing the plurality of glass powder particles of the glass powder layer 21

(2B) a step of forming a plurality of projections 31 on a surface of the mother glass layer 42

(3') a step of forming a phase-separated glass layer 43 including a plurality of projections 32 by subjecting the mother glass layer 42 to phase separation

(4') a step of forming a porous glass layer 200 including a plurality of projections 30 by etching the phase-separated glass layer 43

The step (2A) and the step (2B) may be simultaneously performed or the step (2B) may be performed after the step (2A). When the step (2A) and the step (2B) are simultaneously performed, the plurality of projections 31 can be formed on the surface of the mother glass layer 42 by suitably controlling the composition of the glass powder particles, the particle diameter and particle size distribution of the glass powder particles, and the fusion

conditions (e.g., heat treatment temperature, heat treatment time, and heating rate) of the glass powder particles. For example, by increasing the heating rate, the plurality of projections 31 can be formed on the surface of the mother glass layer 42.

[0108] When the step (2B) is performed after the step (2A), the plurality of projections 31 can be formed on the surface of the mother glass layer 42 by performing dry etching or mechanical polishing. Herein, the projections 31 are projections to be converted into projections 30, on the surface of the porous glass layer 200, in which the width of the projections 30 is larger than the pore size of the pores of the porous glass layer 200 and the distance between the plurality of projections 30 is 75 nm or more and 300 nm or less.

[0109] As described above, when a plurality of projections 31 are formed on the surface of the mother glass layer 42 before the phase separation treatment, the projections 30 are formed on the surface of the porous glass layer 200 by only performing a typical phase separation treatment and etching treatment.

[0110] This embodiment can be combined with the first embodiment.

Examples

[0111] Examples will be described below, but the present

invention is not limited to Examples.

Preparation example of glass powder particles

[0112] A mixed powder of quartz powder, boron oxide, sodium oxide, and alumina having a composition of SiO₂ 63 wt%, B₂O₃ 27 wt%, Na₂O 7 wt%, and Al₂O₃ 3 wt% was melted in a platinum crucible at 1500°C for 24 hours. The resulting glass was cooled to 1300°C and was poured into a graphite mold. The glass was cooled in the air for about 20 minutes, placed in a lehr at 500°C for 5 hours, and then cooled for 24 hours to obtain a glass body. The glass body was crushed with a jet mill until the average particle diameter of the particles reached 2.1 μm to prepare glass powder particles.

Preparation example of glass paste

[0113]

Glass powder particles prepared above: 60.0 parts by mass

α-terpineol: 44.0 parts by mass

Ethylcellulose (registered trademark, ETHOCEL Std 200 (manufactured by The Dow Chemical Company)): 2.0 parts by mass

These raw materials were mixed under stirring to prepare a glass paste.

Example 1

[0114] In Example 1, a structure including a porous layer on a base was produced as follow. The glass paste was

applied by screen printing onto a 50 mm × 50 mm quartz base having a thickness of 0.5 mm (manufactured by Iiyama Precision Glass Co., Ltd.). A printer MT-320TV manufactured by Micro-tec Co., Ltd. was used. A #500 30 mm × 30 mm solid image was used as a screen printing plate.

[0115] Subsequently, the quartz base with the glass paste was placed in a drying furnace at 100°C for 10 minutes to evaporate the solvent, thereby forming a glass powder layer on the base. In a heat treatment process 1, the glass powder layer was heated to 1000°C at a heating rate of 20 °C/min, heat-treated for 5 minutes, and cooled to normal temperature at a cooling rate of 20 °C/min to obtain a mother glass layer on the base. As a result of visual observation of the mother glass layer, the glass powder layer was sufficiently fused and was a transparent layer.

[0116] In the subsequent heat treatment process 2, the mother glass layer was heated to 600°C at a heating rate of 20 °C/min, heat-treated for 50 hours, and cooled to normal temperature at a cooling rate of 20 °C/min to obtain a phase-separated glass layer on the base. The outermost surface of the phase-separated glass layer was then polished.

[0117] The stacked body of the base and the phase-separated glass layer was immersed into a 1.0 mol/L aqueous nitric acid solution heated to 95°C and left to stand at 95°C for 24 hours. The stacked body was then immersed in

distilled water heated to 95°C and left to stand for 3 hours. The stacked body was extracted from the solution and dried at room temperature (20°C) for 12 hours to obtain an optical member 1. It was confirmed from the observation of the optical member 1 that a porous structure having a pore size S_b of 31 nm and a skeleton size L_b of 28 nm was formed on the base. It was also confirmed from a cross-sectional image (Fig. 8) observed using an AFM that projections were formed on the surface of the optical member 1. The distance S_a between the projections was 93 nm and the width L_a of the projections was 67 nm. Pores of the porous structure were observed on the surface of the optical member 1.

Examples 2 and 3

[0118] In Examples 2 and 3, optical members 2 and 3 were respectively produced by conducting the same processes as in Example 1, except that the production conditions were changed to the conditions listed in Table 1.

Comparative Example 1

[0119] In Comparative Example 1, an optical member 4 was produced by conducting the same processes as in Example 1, except that the production conditions were changed to the conditions listed in Table 1. Referring to a cross-sectional image (Fig. 9) of the optical member 4, projections were not observed on the surface of the porous layer.

[0120] Table 2 shows the properties of the porous glass layers of the optical members in Examples 1 to 3 and Comparative Example 1.

[0121]

[Table 1]

		Example 1	Example 2	Example 3	Comparative Example 1
Heat treatment process 1	Temperature (°C)	1000	1000	1000	1000
	Time (min)	5	5	5	5
	Heating rate (°C/min)	20	50	50	20
Heat treatment process 2	Temperature (°C)	600	600	550	600
	Time (hour)	50	50	50	50
	Heating rate (°C/min)	20	20	20	20
Etching process	Temperature (°C)	95	95	80	80
	Time (hour)	24	24	24	24

[0122]

[Table 2]

		Example 1	Example 2	Example 3	Comparative Example 1
Porous glass layer	Porosity (%)	38	42	35	36
	Distance Sa between projections (nm)	93	211	84	62
	Width La of projections (nm)	67	105	138	222
	La/Sa	0.72	0.50	1.64	3.58
	Pore size Sb (nm)	31	32	14	18
	Skeleton size Lb (nm)	28	27	20	30
	Thickness (μm)	2.4	2.6	2.3	2.3

Evaluation

[0123] The following evaluations were performed for the optical members in Examples 1 to 3 and Comparative Example 1 and the quartz base used in Examples 1 to 3 and Comparative Example 1. Table 3 shows the results.

Evaluation of adhesive force

[0124] The adhesive force was measured with an AFM (E-

Sweep manufactured by Seiko Instruments Inc.). A cantilever (force model AFM probe cantilever: FM, manufactured by sQUBE) on which a polystyrene particle having a diameter of 6.1 μm is mounted was attached to the AFM, and measurement was conducted. A point at which the cantilever contacted a sample was assumed to be zero, and a scanner to which the sample was attached was lifted up by 200 nm to press the cantilever against the sample. The adhesive force was determined from a force curve observed when the cantilever was detached from the sample. In each measurement, 20 points were measured and the average of the measured adhesive forces was defined as an adhesive force exerted between the sample and the polystyrene particle. The measurement was performed at 25°C and a humidity of 45%.

[0125] The adhesive force was expressed as an adhesive force index, which was a relative value when the adhesive force of a fluorine-coated glass base serving as a standard sample was assumed to be 1.00. The fluorine-coated glass base was prepared by depositing an evaporation material OF-SR manufactured by Canon Opttron. Inc. on a flat glass base to form a film having a thickness of about 3 nm to 6 nm.

[0126] As is clear from Table 3, the adhesive force indices in Examples 1 to 3 were lower than the adhesive force index in Comparative Example 1.

Evaluation of surface reflectance

[0127] The surface reflectances of the optical members in Examples 1 to 3 and Comparative Example 1 and the quartz base used in Examples 1 to 3 and Comparative Example 1 were measured in a wavelength range of 450 nm to 650 nm in increments of 1 nm with a Lens Spectral Reflectivity Measurement Device (USPM-RU manufactured by Olympus Corporation).

[0128] Fig. 10 shows the results of the surface reflectances. The surface reflectance of the quartz base was about 3.3% in a wavelength range of 450 nm to 650 nm. On the other hand, the surface reflectances of the optical members in Examples 1 to 3 and Comparative Example 1 were 0.5% or less in the wavelength range. Furthermore, the surface reflectances of the optical members in Examples 1 to 3 were lower than that of the optical member in Comparative Example 1.

[0129]

[Table 3]

	Example 1	Example 2	Example 3	Comparative Example 1	Quartz base
Adhesive force index	0.06	0.02	0.21	0.75	3.00
Average reflectance (%)	0.2	0.2	0.3	0.4	3.3

[0130] While the present invention has been described with reference to exemplary embodiments, it is to be understood that the invention is not limited to the disclosed exemplary embodiments. The scope of the following claims is to be

accorded the broadest interpretation so as to encompass all such modifications and equivalent structures and functions.

[0131] This application claims the benefit of Japanese Patent Application No. 2013-136158, filed June 28, 2013, which is hereby incorporated by reference herein in its entirety.

Reference Signs List

[0132] 1 base

10 pore

20 porous layer

203 optical member

CLAIMS

[1] An optical member comprising:

a base; and

a porous layer disposed on the base and comprising a plurality of pores,

wherein a plurality of projections are formed on a surface of the porous layer,

a width of the projections is larger than a pore size of the pores of the porous layer, and

a distance between the projections is 75 nm or more and 300 nm or less.

[2] The optical member according to Claim 1, wherein the width of the projections is 5 nm or more and 500 nm or less.

[3] The optical member according to Claim 1 or 2, wherein a ratio of the width of the projections to the distance between the projections is less than 1.00.

[4] The optical member according to Claim 3, wherein the ratio of the width of the projections to the distance between the projections is less than 0.60.

[5] The optical member according to any one of Claims 1 to 4, wherein the pores are present on the surface of the porous layer.

[6] The optical member according to any one of Claims 1 to 5, wherein the porous layer is a porous silica layer.

[7] The optical member according to any one of Claims 1 to

6, wherein the porous layer is a porous glass layer.

[8] An image pickup apparatus comprising:

the optical member according to any one of Claims 1 to 7; and

an image pickup element.

[9] A method for producing an optical member comprising a base and a porous layer disposed on the base and comprising a plurality of pores, the method comprising the steps of:

forming a porous layer comprising a plurality of pores on a base; and

forming a plurality of projections on a surface of the porous layer,

wherein a width of the projections is larger than a pore size of the pores of the porous layer, and

a distance between the projections is 75 nm or more and 300 nm or less.

[10] The method for producing an optical member according to Claim 9,

wherein the porous layer is a porous glass layer, and the step of forming a porous layer comprises the steps of:

forming a glass powder layer containing a plurality of glass powder particles on a base;

forming a mother glass layer by fusing the plurality of glass powder particles of the glass powder

layer;

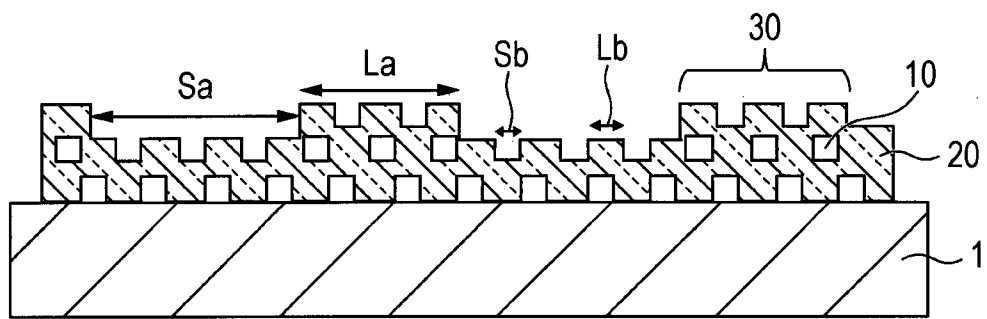
forming a phase-separated glass layer by subjecting the mother glass layer to phase separation; and

forming a porous glass layer by etching the phase-separated glass layer.

[11] The method for producing an optical member according to Claim 10, wherein the step of forming a plurality of projections on a surface of the porous layer and the step of forming a porous glass layer are simultaneously performed.

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FIG. 1



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FIG. 2A

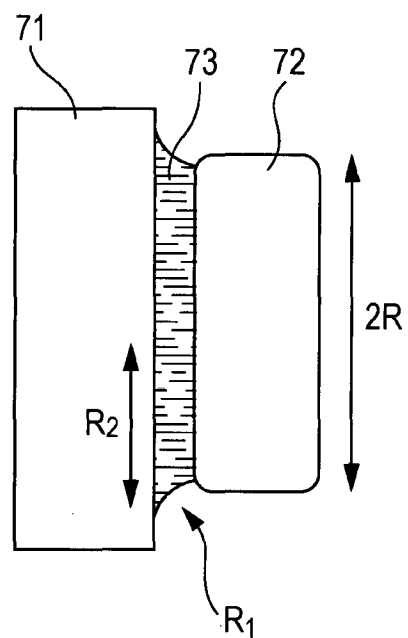
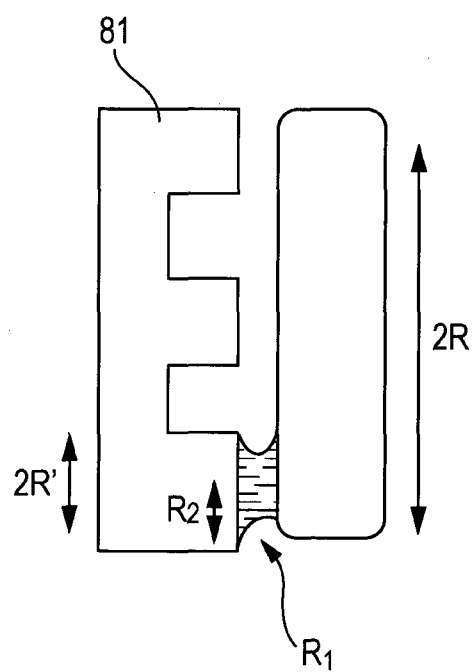
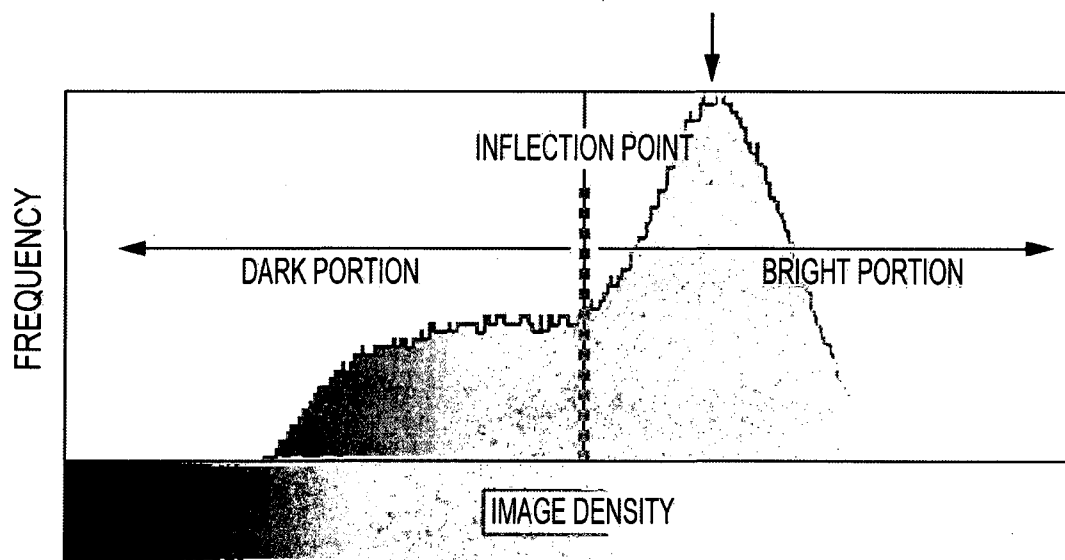


FIG. 2B



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FIG. 3



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FIG. 4A

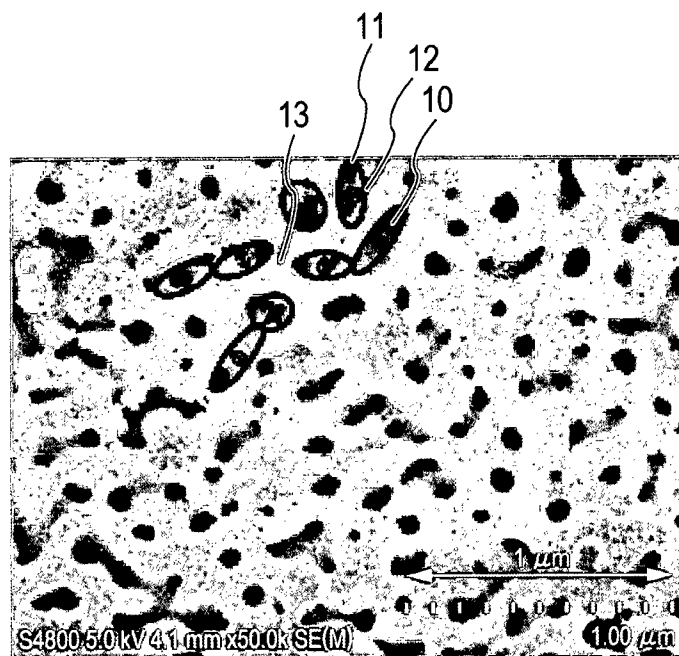
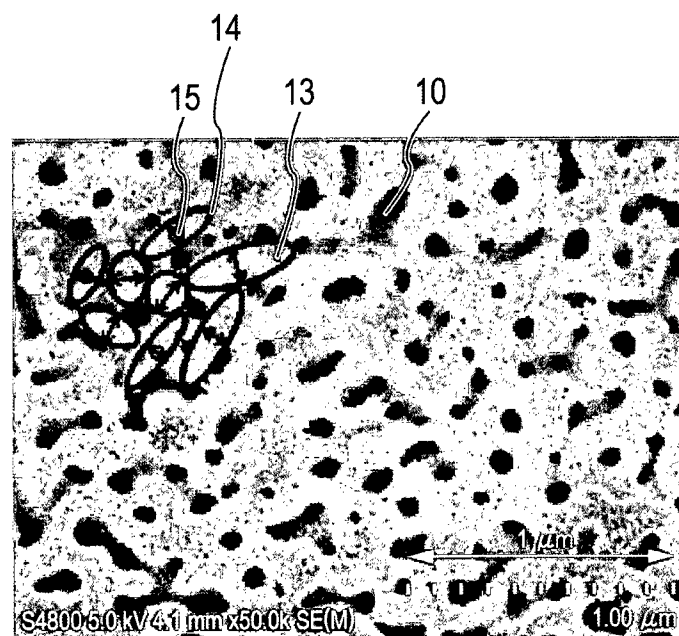
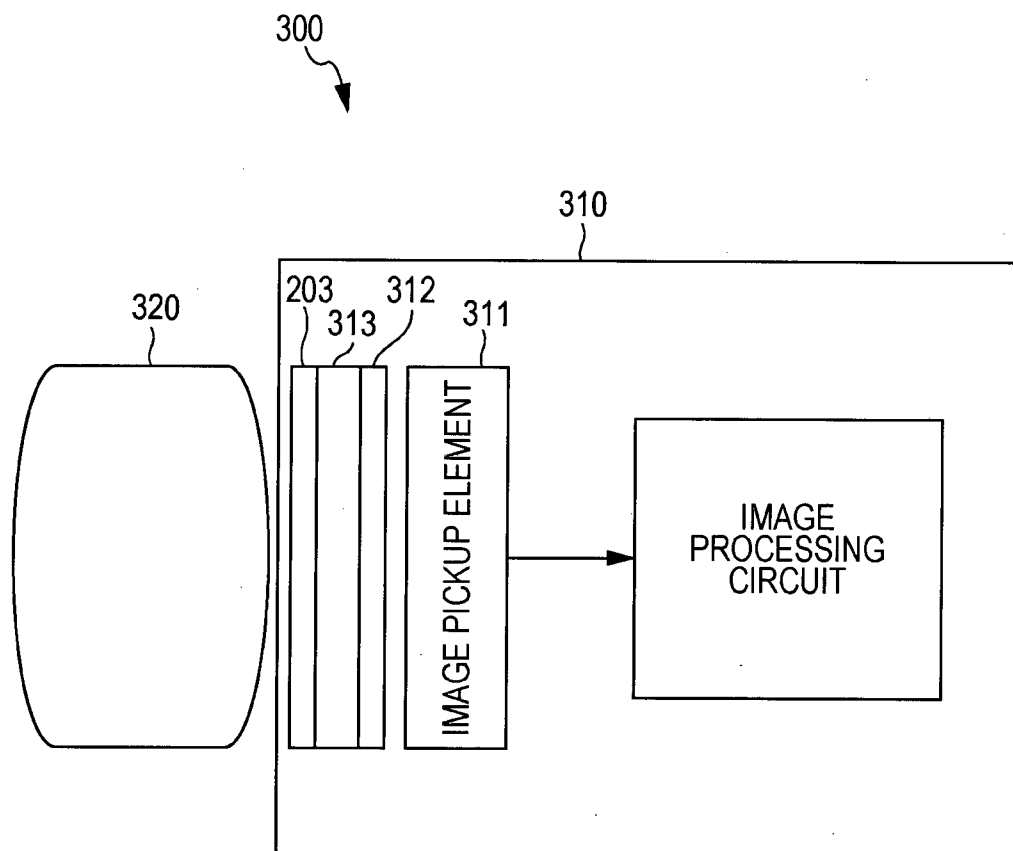


FIG. 4B



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FIG. 5



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FIG. 6A

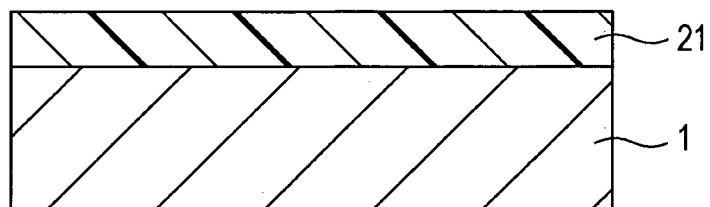


FIG. 6B

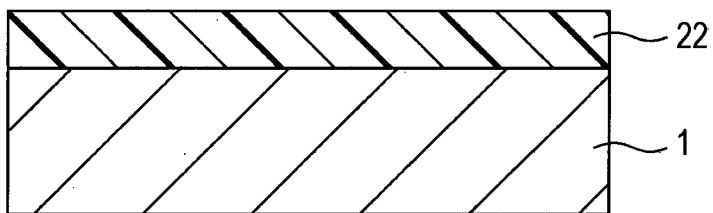


FIG. 6C

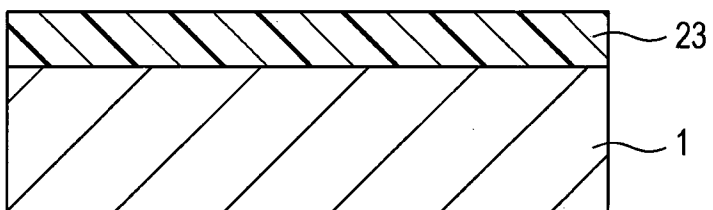


FIG. 6D

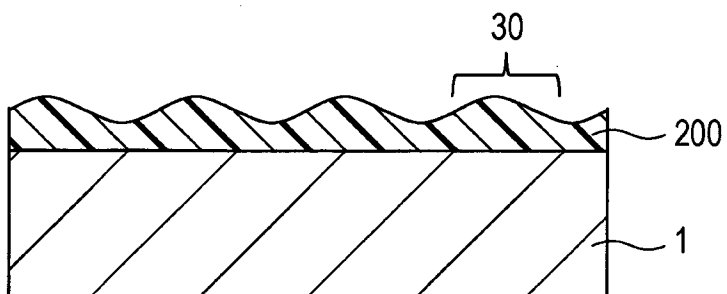


FIG. 7A

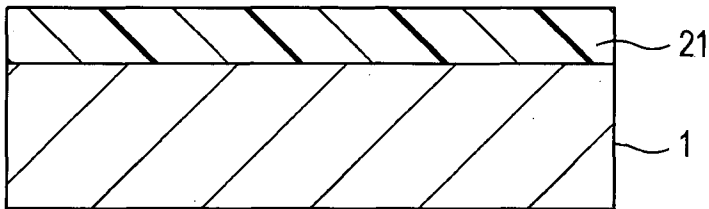


FIG. 7B

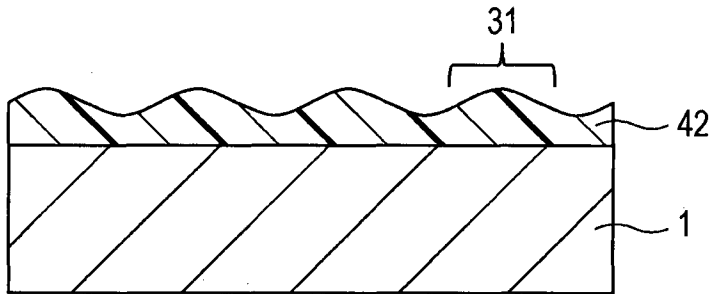


FIG. 7C

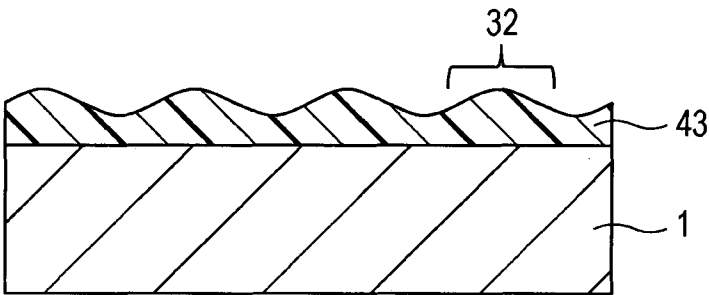
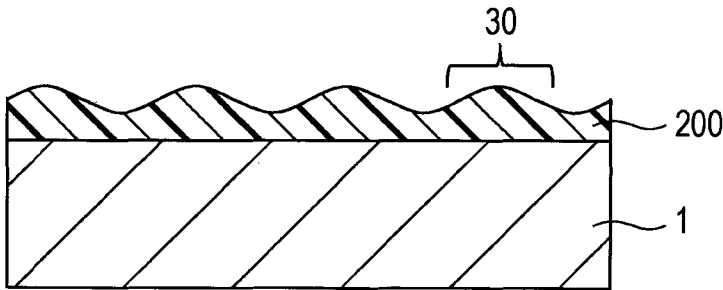
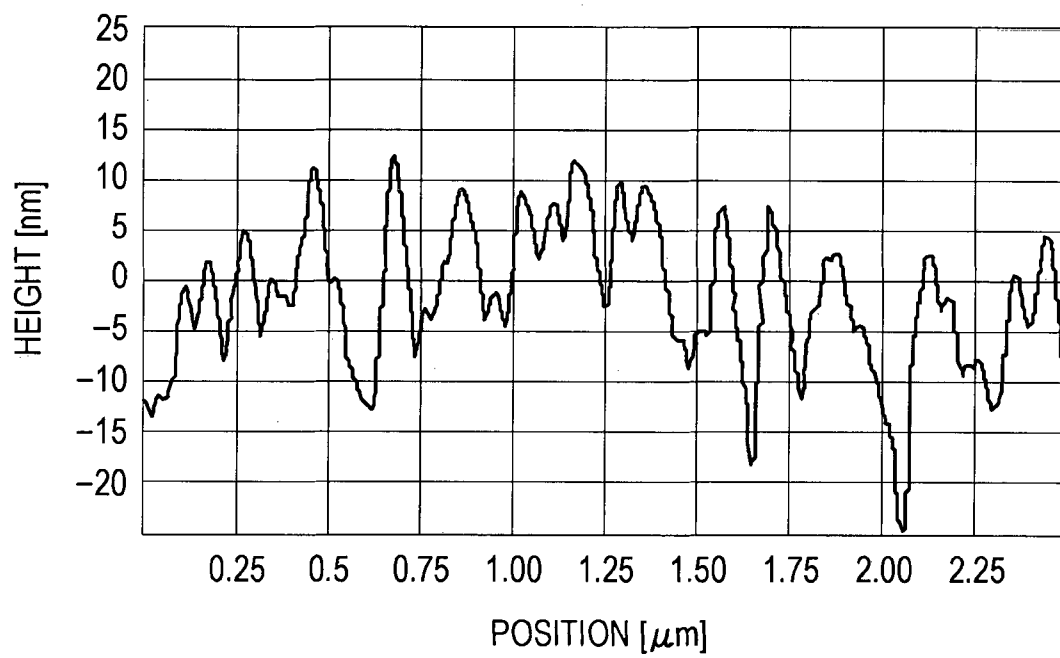


FIG. 7D



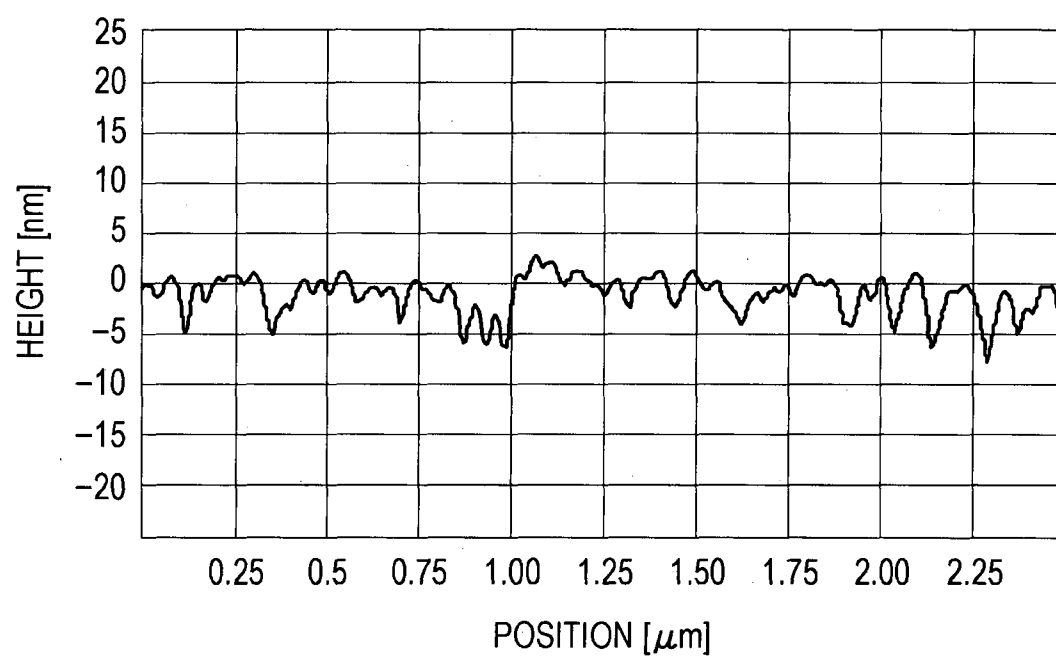
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FIG. 8



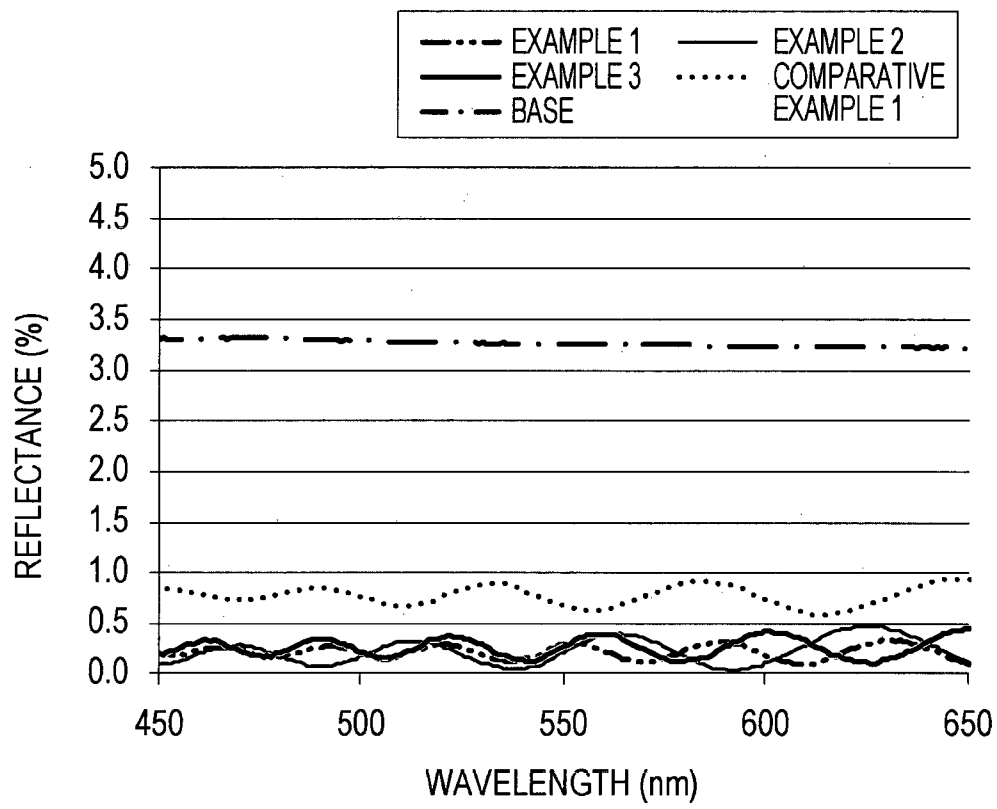
9/10

FIG. 9



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FIG. 10



INTERNATIONAL SEARCH REPORT

International application No

PCT/JP2014/064554

A. CLASSIFICATION OF SUBJECT MATTER
 INV. C03C17/00 G02B1/11 G02B1/10
 ADD.

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)
 C03C G02B

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

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

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2 536 764 A (MOULTON HAROLD R) 2 January 1951 (1951-01-02) column 3, line 32 - line 36 figure 1	1-6,8,9
X	----- WO 2013/088632 A1 (CANON KK [JP]) 20 June 2013 (2013-06-20) page 17, paragraph 101 - paragraph 106; example 1	1-11
A	----- JP 2011 221273 A (SEIKO EPSON CORP) 4 November 2011 (2011-11-04) abstract -----	1-11



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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Information on patent family members

International application No

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