perpendicular to the film-formation surface F of the base 2, in a visible range and an adjacent range, according to Example 1.

The graphs indicate that, in Example 1, low reflection (for example, not greater than 1% over the entire visible range) was realized for visible light.

[0023]

FIG. 6 shows a graph of both surface reflectance (mainly, a ratio of the total intensity of the reflected light R1 and the reflected light R2 to an intensity of the incident light I1) of light in a visible range and an adjacent range in the case of an incidence angle θ with respect to the film-formation surface F of the base 2 being varied, according to Example 1.

FIG. 7 shows a graph representing incidence angle dependency of both surface reflectance, in which the horizontal axis represents an incidence angle θ and the vertical axis represents an average of reflectances (average reflectance) in a specific range in a visible range. In the description herein, the specific range is a range of not less than 420 nm and not greater than 680 nm. In the following description, all of the various average reflectances were calculated in this specific range.

These graphs indicate that, in Example 1, low reflection (for example, not greater than 2% over the entire visible range) was realized to almost the same degree as in perpendicular incidence, up to the incidence angle θ=45°, and low reflection was realized to such a degree that the average-reflectance in the visible range was not greater than 2% also at the incidence angle θ=50°. That is, the low reflection in Example 1 was realized in a wide range of incidence angles θ (characteristics of moth-eye structure), and incidence angle dependency of low reflection in Example 1 can be said to be low in a range of not less than 0° and not greater than 50°.

[0024]

Furthermore, a structure and a component of the optical film 4 of Example 1 were observed in the following procedure.

That is, the optical film 4 was formed on one surface of a substrate made of PC in the same manufacturing method as in Example 1, and the optical film 4 on the substrate was cut by Ga (gallium) beams into such a size as to be stored in a sample holder made of copper (FIB (focused ion beam) process). The substrate having the optical film 4 was stored in the sample holder, and the cut substrate having the optical film 4 was covered with a carbon protective film in order to retain the structure of the optical film 4, to produce an observation target.

The observation target was observed by using a transmission electron microscope

(TEM), and characteristic X-rays were applied to the observation target to analyze elements of the optical film 4.

[0025]

FIG. 8 shows a graph of a spectrum by characteristic X-rays.

FIG. 8 indicates that the observation target contained C (carbon atom), O (oxygen atom), Cu (copper), Ga (gallium), Al (aluminium atom), and Si (silicon atom).

Among them, Cu was derived from the sample holder. Ga was derived from FIB process. Furthermore, C was derived from the protective film. Therefore, the optical film 4 contained O, Al, and Si.

[0026]

FIG. 9 shows an observed image by the TEM. FIG. 10 shows an image (C-Kα rays overlap image) in which the pixel concentration is proportionally enhanced such that the higher the intensity at the position of a pixel is, the higher the concentration of the pixel is, according to the distribution of the intensity of Kα rays of C, in the observation range in FIG. 9. FIG. 11 shows an image (O-Kα rays overlap image) similar to that in FIG. 10, for Kα rays of O. FIG. 12 shows an image (Al-Kα rays overlap image) similar to that in FIG. 10, for Kα rays of Al. FIG. 13 shows an image (Si-Kα rays overlap image) similar to that in FIG. 10, for Kα rays of Si.

These figures indicate that a layer was formed on the substrate (laterally elongated rectangular portion occupying the lowermost portion in the image), and the minutely uneven structure was formed on the layer. It is indicated that C was in a portion (corresponding to the protective film) other than the substrate, the layer, and the minutely uneven structure. It is further indicated that O was in the layer and the minutely uneven structure. Furthermore, it is indicated that Al was in the layer on the substrate. In addition, it is indicated that Si was in the minutely uneven structure.

[0027]

According to the above-described observed matters being collectively considered as appropriate, the main component of the layer on the substrate was an oxide of Al, and the main component of the minutely uneven structure was an oxide of Si. Furthermore, by taking into consideration the other observed matters such as stability thereof, it can be said that the main component of the layer on the substrate was Al_2O_3, and the main component of the minutely uneven structure was SiO_2.

By taking into consideration the observation results of, for example, Examples 2 to 38 described below as appropriate, it can be said that the Al_2O_3 layer (layer on the substrate side) on

the substrate and the SiO_2 layer (uneven layer) having the minutely uneven structure were clearly divided into two layers in some cases, and the component was gradually changed according to the position in the film thickness direction (direction perpendicular to the film) without strictly dividing the Al_2O_3 layer and the SiO_2 layer into two layers in other cases, according to differences in the material and film thickness of the Al-based manufacture intermediate film 22, and various manufacturing conditions such as a temperature of the solution SL.

In the latter case, the boundary between various films may be unclear. Furthermore, in this case, typically, in the layer on the substrate side, a component ratio of Al_2O_3 is higher at a position closer to the substrate, and a component ratio of SiO_2 to Al_2O_3 is higher at a position farther from the substrate. That is, in the film thickness direction, the composition ratio of SiO_2 to Al_2O_3 is proportional to the distance from the substrate.

Furthermore, the uneven layer can contain Al_2O_3 in a state where the Al_2O_3 does not become a main component. For example, the uneven layer can have a core (skeleton), of the minutely uneven structure, which contains Al_2O_3 as a main component, and a coating which contains, as a main component, SiO_2 covering a part or the entirety of the core.

[0028]

[Examples 2 to 37 and Comparative example 1]

<<Manufacturing of Examples 2 to 37 and the like>>

Examples 2 to 37 and Comparative example 1 were manufactured similarly to Example 1. However, as indicated below in Table 2 to Table 9, in each of Examples 2 to 37 and Comparative example 1, at least one of the concentration of silica in the solution SL, a material of the Al-based manufacture intermediate film 22, a material of the base 2, a temperature of the solution SL, and a physical film thickness of the Al-based manufacture intermediate film 22 was different from that in Example 1.

All of the Al-based manufacture intermediate films 22 of Examples 2 to 37 and Comparative example 1 were formed by DC sputtering, and the process condition thereof was set for each material of the Al-based manufacture intermediate film 22 as indicated above in Table 1. In Table 1, process conditions for vapor deposition are also indicated as modifications.

[0029]

[Table 2]

	Example 1	Example 2	Example 3	Example 4	Example 5
Concentration (mg/L) of silica	0.06	0.06	0.06	0.06	0.06
Material of Al-based	AlN	AlN	AlN	AlN	AlN

Temperature (°C) of solution	80	90	90	90	90
Thickness (nm) of Al-based intermediate film	75	78.5	78.5	78.5	78.5
Average reflectance (%)	3.82	0.27	0.41	0.5	0.75

[Table 9]

	Example 36	Example 37	Comparative example 1
Concentration (mg/L) of silica	2	10	20
Material of Al-based intermediate film	AlN	AlN	AlN
Material of substrate	PC	PC	PC
Temperature (°C) of solution	90	90	90
Thickness (nm) of Al-based intermediate film	78.5	78.5	78.5
Average reflectance (%)	1.35	4.38	9.82

[0032]

<<Characteristics and the like of Examples 2 to 37 and Comparative example 1>>

FIG. 14 shows a graph of one surface reflectance of light in a visible range and an adjacent range in perpendicular incidence, according to Examples 2 to 6. FIG. 15 shows a graph similar to that in FIG. 14, for Examples 7 to 11. FIG. 16 shows a graph similar to that in FIG. 14, for Examples 12 to 16. FIG. 17 shows a graph similar to that in FIG. 14, for Examples 17 to 20. FIG. 18 shows a graph similar to that in FIG. 14, for Examples 21 to 26. FIG. 19 shows a graph similar to that in FIG. 14, for Examples 27 to 31. FIG. 20 shows a graph similar to that in FIG. 14, for Examples 32 to 37 and Comparative example 1.

In the lowermost lines in Table 2 to Table 9, an average reflectance in each example is indicated.

According to these graphs and tables, in comparative example 1, the average reflectance was greater than 9% and was almost 10%, and it cannot be said that reflection of visible light was sufficiently inhibited.

Meanwhile, in Examples 2 to 37, similarly to Example 1, it is indicated that low reflection of visible light was realized.

According to various observations, also in Examples 2 to 37, similarly to Example 1, it was confirmed that low reflection was realized in a state where incidence angle dependency was

low, and the minutely uneven structure was made of SiO_2 (the main component of the minutely uneven structure was a SiO_2 layer), and, furthermore, a layer made of Al_2O_3 (having Al_2O_3 as a main component) was formed between the minutely uneven structure and the substrate.

Particularly, in a case where the Al-based manufacture intermediate film 22 was made of AlN, the optical film 4 for antireflection was formed with various physical film thicknesses of the Al-based manufacture intermediate films 22 and various temperatures of the solution SL (Examples 1 to 20).

Furthermore, also in a case where the Al-based manufacture intermediate film 22 was made of Al_2O_3 (Examples 23 to 28, 30 to 31) or Al (Example 29), the optical film 4 for antireflection was formed.

Moreover, even when the substrate was a white sheet glass, the optical film 4 for antireflection was formed (Examples 21 to 29).

[0033]

<<Temperature of solution and the like · Examples 2, 7, 10, 15, and 18>>

Change of characteristics according to change of temperature of the solution SL was found by comparison among the above-described Examples 2, 7, 10, 15, and 18.

Examples 2, 7, 10, 15, and 18 were the same in the concentration (0.06 mg/l) of silica, the material (AlN) of the Al-based manufacture intermediate film 22, the material (PC) of the substrate, and the physical film thickness (78.5 to 78.51 nm) of the Al-based manufacture intermediate film 22, and were different in the temperature of the solution SL, and the temperatures of the solution SL in Examples 2, 7, 10, 15, and 18 were 95, 90, 85, 80, and 75°C, respectively.

[0034]

FIG. 21 shows a graph for one surface reflectance in perpendicular incidence in Examples 2, 7, 10, 15, and 18.

FIG. 22 shows a graph for the average reflectance in Examples 2, 7, 10, 15, and 18.

According to these figures, in a case where the temperature of the solution SL was not lower than 80°C, one surface reflectance was further reduced as compared with a case where the temperature of the solution SL was lower than 80°C (Example 18).

[0035]

<<Concentration of silica in solution and the like · Examples 32 to 37, Comparative example 1>>

Change of characteristics according to change of concentration of silica in solution SL

was found by comparison among the above-described Examples 32 to 37 and Comparative example 1.

Examples 32 to 37 and Comparative example 1 were the same in the material (AlN) of the Al-based manufacture intermediate film 22, the material (PC) of the substrate, the temperature (90°C) of the solution SL, and the physical film thickness (78.5 nm) of the Al-based manufacture intermediate film 22, and were different in the concentration of silica in the solution SL, and the concentrations of silica in the solution SL in Examples 32 to 37 and Comparative example 1 were 0.003, 0.06, 0.5, 1, 2, 10, and 20 mg/l, respectively.

The concentration of silica in the solution SL was adjusted by using pure water (Example 32) having an extremely high purity or pure water (Example 33) having about normal purity, as it was, or the concentration of silica in the solution SL was adjusted by putting an appropriate amount of silica gel into the latter pure water and sufficiently stirring the mixture. In the former pure water and the latter pure water, a small amount of silica was not completely eliminated and was left.

The concentration of silica was obtained in the manner as described in Example 1.

[0036]

The above-described FIG. 20 exactly shows Examples 32 to 37 and Comparative example 1.

FIG. 23 shows a graph representing a relationship between the average reflectance (vertical axis) and the concentration (horizontal axis) of silica in the solution SL, in Examples 32 to 37 and Comparative example 1.

Table 10 indicates a relationship between the concentration of silica in the solution SL and the average reflectance in Examples 32 to 37 and Comparative example 1.

[0037]

[Table 10]

Concentration (mg/L) of silica	0.003	0.06	0.5	1	2	10	20
Average reflectance (%) at 420 to 680 nm	0.36	0.33	0.4	0.52	0.75	3.46	15.63

[0038]

According to these figures and tables, in a case where the concentration of silica in solution SL was not greater than 10 mg/l, one surface reflectance was further reduced as compared with a case (Comparative Example 1) where the concentration of silica in solution SL was greater than 10 mg/l. In a case where the temperature of the solution SL was not greater than 2 mg/l, one surface reflectance was further reduced as compared with a case (Example 37, Comparative

CLAIMS

[Claim 1] An optical product manufacturing method comprising:

forming, on a base, an Al-based manufacture intermediate film made of aluminium, an aluminium alloy, or an aluminium compound; and

immersing the base having the Al-based manufacture intermediate film in an aqueous solution of silica, wherein

the silica in the aqueous solution has a concentration of not greater than 10 mg/l, and

the aqueous solution has a temperature of not lower than 75°C and not higher than 98°C.

[Claim 2] The optical product manufacturing method according to claim 1, wherein the Al-based manufacture intermediate film is made of at least one of Al, Al_2O_3, AlN, and AlON.

[Claim 3] The optical product manufacturing method according to claim 1 or claim 2, wherein

the Al-based manufacture intermediate film is changed to an optical film by immersing the base having the Al-based manufacture intermediate film in the aqueous solution, and

the optical film has

an Al_2O_3 layer that is disposed on the base side and that contains Al_2O_3 such that a volume ratio of Al_2O_3 is greater than half an entire volume ratio and

an SiO_2 layer that has a minutely uneven structure and that contains SiO_2 such that a volume ratio of SiO_2 is greater than half an entire volume ratio.

[Claim 4] The optical product manufacturing method according to claim 3, wherein the minutely uneven structure is at least one of an irregular fluff-like structure, an irregular multiple-pyramids-shaped structure, and an irregular flower-frog-shaped structure.