

DESCRIPTION

SILVER-ALLOY SPUTTERING TARGET FOR CONDUCTIVE-FILM FORMATION,
AND METHOD FOR PRODUCING SAME

5

TECHNICAL FIELD

[0001]

The present invention relates to a silver-alloy sputtering target for
conductive-film formation (for forming a conductive film) such as a reflective electrode in
10 an organic EL element, a wiring film in a touch panel, and the like, and a method for
producing the same.

The present application claims priority on Japanese Patent Application No.
2012-002072 filed on January 10, 2012, the content of which is incorporated herein by
reference.

15

BACKGROUND ART

[0002]

In an organic EL element, holes and electrons are injected into an organic EL film
from an anode and a cathode respectively by applying a voltage between the anode and the
20 cathode formed on both sides of an organic EL light-emitting layer. Then, light is
emitted when the holes and the electrons are combined together in the organic EL
light-emitting layer. The organic EL element is a light-emitting element using the
above-described light-emitting principle, and the organic EL element has been attracting
significant attention in recent years for use in display devices. As a driving method of
25 the organic EL element, there are a passive matrix and an active matrix. In the active

matrix, one or more thin film transistors are provided in a pixel, and thus high-speed switching is possible. Therefore, the active matrix becomes advantageous in terms of an increase in the contrast ratio and an increase in the definition, and is a driving method capable of exhibiting the characteristics of the organic EL element.

5 In addition, as a light extraction (emission) system, there are a bottom emission system in which light is emitted from a transparent substrate side and a top emission system in which light is emitted from a side opposite to a substrate, and the top emission system having a high aperture ratio is advantageous in terms of an increase in the brightness.

10 [0003]

A reflective electrode film in a top emission structure desirably has a high reflectivity and strong corrosion resistance so as to efficiently reflect light emitted from the organic EL layer. In addition, the reflective electrode film also desirably has a low resistance as an electrode. An Ag alloy and an Al alloy are known as materials having
15 the above-described characteristics, and the Ag alloy is preferred due to its high reflectivity of visible light so as to obtain an organic EL element having a higher brightness. Here, a sputtering method is employed so as to form the reflective electrode film on the organic EL element, and a silver alloy target is used (Patent Document 1).

[0004]

20 In response to an increase in the size of a glass substrate in the production of the organic EL element, a large silver alloy target has been used to form the reflective electrode film. Here, when a high electric power is supplied to a large target so as to carry out sputtering, a phenomenon called “splash” caused by the abnormal discharge of the target occurs. When this phenomenon occurs, molten fine particles are attached to
25 the substrate such that wires or electrodes may be short-circuited. Thereby, there is a

problem in that the yield of the organic EL element decreases. Since the reflective electrode layer in the organic EL element of the top emission system serves as a base layer for the organic light-emitting layer, there is a demand for greater flatness; and therefore, it is necessary to further suppress the splash.

5 In order to solve the above-described problem, Patent Documents 2 and 3 propose silver alloy targets for forming a reflective electrode film of the organic EL element capable of suppressing the splash even when a high electric power is supplied to the target in response to an increase in the size of the target and a method for producing the same.

[0005]

10 The silver alloy targets for forming a reflective electrode film described in Patent Documents 2 and 3 have enabled the suppression of the splash even when a high electric power is supplied. However, in a large silver alloy target, there is a tendency that the number of times of arc discharging increases as the target is consumed and the splash caused by the arc discharging occurs more frequently; and therefore, there is a demand for
15 further improvement.

In addition, studies are being conducted regarding the use of a silver alloy film in a conductive film such as a lead-out wiring in a touch panel in addition to the reflective electrode film for the organic EL element. However, the use of pure Ag as the wiring film causes migration; and thereby, the possibility of poor short-circuiting increases.

20 Therefore, the employment of a silver alloy film is being studied.

PRIOR ART DOCUMENTS

Patent Documents

[0006]

25 Patent Document1: PCT International Publication No. WO 2002/077317

Patent Document 2: Japanese Unexamined Patent Application, First Publication
No. 2011-100719

Patent Document 3: Japanese Unexamined Patent Application, First Publication
No. 2011-162876

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DISCLOSURE OF THE INVENTION

Problems to be Solved by the Invention

[0007]

The present invention has been made in consideration of the above-described
10 circumstances, and the present invention aims to provide a silver-alloy sputtering target for
conductive-film formation capable of further suppressing arc discharging and the splash
and a method for producing the same.

Means for Solving the Problems

15 [0008]

As a result of intensive studies, the present inventors found out that, in order to
suppress the increase in the number of times of arc discharging in response to the
consumption of a target, it is effective to further refine crystal grains to an average grain
diameter of less than 150 μm and to suppress the dispersion (variation) thereof within 20%
20 or less of the average grain diameter.

Based on the above-described finding, a first aspect of a silver-alloy sputtering
target for conductive-film formation of the invention has a component composition
including In at a content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag
and inevitable impurities, wherein an average grain diameter of crystal grains in an alloy is
25 in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of

the crystal grains is in a range of 20% or less of the average grain diameter.

[0009]

In is solid-solubilized in Ag so as to suppress the crystal grain growth in the target; and therefore, In is effective for refining crystal grains. Since In improves the hardness of the target, In suppresses warpage during machining. In improves the corrosion resistance and the heat resistance of a film formed through sputtering. In the case where the content of In is less than 0.1 mass%, the above-described effects cannot be obtained, and in the case where the content of In exceeds 1.5 mass%, the reflectivity or the electric resistance of the film decreases.

The reason for setting the average grain diameter to be in a range of 30 μm or more to less than 150 μm will be described below. An average grain diameter of less than 30 μm is not realistic and causes an increase in the production costs. In addition, in the case where the average grain diameter is 150 μm or more, the number of times of occurring abnormal discharge tends to greatly increase in response to the consumption of the target during sputtering.

In the case where the dispersion of the average grain diameter exceeds 20%, the number of times of occurring abnormal discharge tends to greatly increase in response to the consumption of the target during sputtering.

[0010]

A second aspect of the silver-alloy sputtering target for conductive-film formation of the invention has a component composition including In and Sn at a total content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag and inevitable impurities, wherein an average grain diameter of crystal grains in an alloy is in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of the crystal grains is in a

range of 20% or less of the average grain diameter.

[0011]

Similarly to In, Sn is solid-solubilized in Ag so as to suppress the crystal grain growth in the target; and therefore, Sn is effective for refining crystal grains. Since In and Sn improve the hardness of the target, In and Sn suppress warpage during machining. In and Sn improve the corrosion resistance and the heat resistance of a film formed through sputtering. In the case where the total content of In and Sn is less than 0.1 mass%, the above-described effects cannot be obtained, and, in the case where the total content of In and Sn exceeds 1.5 mass%, the reflectivity or the electric resistance of the film decreases.

[0012]

A third aspect of the silver-alloy sputtering target for conductive-film formation of the invention has a component composition including: In at a content in a range of 0.1 mass% to 1.5 mass%; and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag and inevitable impurities, wherein an average grain diameter of crystal grains in an alloy is in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.

A fourth aspect of the silver-alloy sputtering target for conductive-film formation of the invention has a component composition including: In and Sn at a total content in a range of 0.1 mass% to 1.5 mass%; and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag and inevitable impurities, wherein an average grain diameter of crystal grains in an alloy is in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of the crystal grains is in a

range of 20% or less of the average grain diameter.

[0013]

Sb and Ga are solid-solubilized in Ag so as to further suppress crystal grain growth. Sb and Ga further improve the corrosion resistance and the heat resistance of a film formed through sputtering. Particularly, Ga improves the resistance against chloridation of the film. In the case where the total content of Sb and Ga is less than 0.1 mass%, the above-described effect cannot be obtained. In the case where the total content of Sb and Ga exceeds 2.5 mass%, not only does the reflectivity or the electric resistance of the film decrease, but also cracking tends to occur during hot rolling.

[0014]

In the first to fourth aspects of the silver-alloy sputtering target for conductive-film formation of the invention, the average grain diameter of the crystal grains may be in a range of less than 120 μm .

In the case where the average grain diameter of the crystal grains is in a range of less than 120 μm , it is possible to further suppress arc discharging and splash.

[0015]

In a first aspect of a method for producing a silver-alloy sputtering target for conductive-film formation of the invention, a cast ingot having a component composition including In at a content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag and inevitable impurities is subjected to a hot rolling step, a cooling step and a machining step sequentially; and thereby, a silver-alloy sputtering target is produced. In the hot rolling step, one or more passes of finish hot rolling are carried out under conditions where a rolling reduction rate per pass is in a range of 20% to 50%, a strain rate is in a range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of 400°C to 650°C, and in the cooling step, quenching is carried out at a cooling rate in a range of 200 °C/min

to 1000 °C/min.

[0016]

In a second aspect of a method for producing a silver-alloy sputtering target for
conductive-film formation of the invention, a cast ingot having a component composition
5 including In and Sn at a total content in a range of 0.1 mass% to 1.5 mass% with a
remainder of Ag and inevitable impurities is subjected to a hot rolling step, a cooling step,
and a machining step sequentially; and thereby, a silver-alloy sputtering target is produced.
In the hot rolling step, one or more passes of finish hot rolling are carried out under
conditions where a rolling reduction rate per pass is in a range of 20% to 50%, a strain rate
10 is in a range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of 400°C to
650°C, and in the cooling step, quenching is carried out at a cooling rate in a range of 200
°C/min to 1000 °C/min.

[0017]

In a third aspect of a method for producing a silver-alloy sputtering target for
15 conductive-film formation of the invention, a cast ingot having a component composition
including: In at a content in a range of 0.1 mass% to 1.5 mass%; and either one or both of
Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag
and inevitable impurities is subjected to a hot rolling step, a cooling step, and a machining
step sequentially; and thereby, a silver-alloy sputtering target is produced. In the hot
20 rolling step, one or more passes of finish hot rolling are carried out under conditions
where a rolling reduction rate per pass is in a range of 20% to 50%, a strain rate is in a
range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of 400°C to 650°C,
and in the cooling step, quenching is carried out at a cooling rate in a range of 200 °C/min
to 1000 °C/min.

In a fourth aspect of a method for producing a silver-alloy sputtering target for conductive-film formation of the invention, a cast ingot having a component composition including: In and Sn at a total content in a range of 0.1 mass% to 1.5 mass%; and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag and inevitable impurities is subjected to a hot rolling step, a cooling step, and a machining step sequentially; and thereby, a silver-alloy sputtering target is produced. In the hot rolling step, one or more passes of finish hot rolling are carried out under conditions where a rolling reduction rate per pass is in a range of 20% to 50%, a strain rate is in a range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of 400°C to 650°C, and in the cooling step, quenching is carried out at a cooling rate in a range of 200 °C/min to 1000 °C/min.

[0018]

The reason for setting the rolling reduction rate per pass of the finish hot rolling to be in a range of 20% to 50% will be described. In the case where the rolling reduction rate is less than 20%, crystal grains fail to be sufficiently refined. When an attempt is made to obtain a rolling reduction rate of more than 50%, the load of a rolling machine becomes excessive, which is not realistic.

In addition, the reason for setting the strain rate to be in a range of 3 /sec to 15 /sec will be described below. In the case where the strain rate is less than 3 /sec, crystal grains fail to be sufficiently refined, and mixed grains of fine grains and coarse grains tend to be generated. The strain rate exceeding 15 /sec makes the load of the rolling machine excessive; and therefore, this strain rate is not realistic.

In the case where the temperature after each pass is lower than 400°C, dynamic recrystallization becomes insufficient; and thereby, the dispersion of the crystal grain

diameter tends to become greater. In the case where the temperature after each pass exceeds 650°C, crystal grains grow such that the average crystal grain diameter reaches 150 μm or more.

In addition, the quenching after the hot rolling suppresses the growth of the crystal grains; and thereby, a target having fine crystal grains can be obtained. In the case where a cooling rate is less than 200 °C/min, the effect of suppressing the growth of crystal grains is not sufficient. Even in the case where the cooling rate exceeds 1000 °C/min, there is no contribution to additional refinement.

10 Effects of the Invention

[0019]

According to the aspects of the invention, it is possible to obtain a target capable of further suppressing arc discharging and the splash even when a high electric power is supplied during sputtering, and it is possible to obtain a conductive film having a high reflectivity and excellent durability by sputtering the above-described target.

EMBODIMENTS OF THE INVENTION

[0020]

Hereinafter, an embodiment of a silver-alloy sputtering target for conductive-film formation (for forming a conductive film) of the invention and a method for producing the same will be described. Meanwhile, “%” represents mass% unless particularly otherwise described or % represents the intrinsic unit of a numeric value.

[0021]

In the target, the target surface (surface being subjected to sputtering of the target)

has an area of 0.25 m^2 or more. In the case of a rectangular target, at least one side is 500 mm or more long, and the upper limit of the length is preferably 3000 mm from the viewpoint of the handling of the target. Meanwhile, the upper limit of the width is preferably 1700 mm from the viewpoint of the upper limit of the size that can be rolled in an ordinary rolling machine used in a hot rolling step. In addition, the thickness of the target is preferably in a range of 6 mm or more from the viewpoint of the exchange frequency of the target, and the thickness is preferably in a range of 25 mm or less from the viewpoint of the discharging stability of magnetron sputtering.

[0022]

A silver-alloy sputtering target for conductive-film formation of a first embodiment is made of a silver alloy having a component composition including In at a content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag and inevitable impurities. The average grain diameter of crystal grains in the alloy is in a range of $30 \mu\text{m}$ or more to less than $150 \mu\text{m}$, and the dispersion of the grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.

[0023]

Ag has an effect of conferring a high reflectivity and a low resistance to a reflective electrode film in an organic EL element and a wiring film in a touch panel that are formed through sputtering.

[0024]

In improves the hardness of the target; and therefore, In suppresses warpage during machining. Particularly, it is possible to suppress warpage during the machining of a large target which includes a target surface having an area of 0.25 m^2 or more. In addition, In has an effect of improving the corrosion resistance and the heat resistance of a reflective electrode film in an organic EL element formed through sputtering. The

above-described effects are brought about by the following action. In refines crystal grains in a film, and In also decreases the surface roughness of the film. In addition, In is solid-solubilized in Ag so as to increase the strength of the crystal grains; and thereby, the coarsening of the crystal grains due to heat is suppressed. Therefore, In has effects of suppressing an increase in the surface roughness of a film and suppressing a decrease in the reflectivity due to corrosion of a film. Therefore, in a reflective electrode film or a wiring film formed using the silver-alloy sputtering target for conductive-film formation, the corrosion resistance and the heat resistance of the film are improved. Therefore, the silver-alloy sputtering target for conductive-film formation contributes to an increase in the brightness of an organic EL element and the reliability of wiring in a touch panel and the like.

The reason for limiting the content of In in the above-described range will be described. In the case where the content of In is less than 0.1 mass%, the above-described effects of the addition of In cannot be obtained. In the case where the content of In exceeds 1.5 mass%, the electric resistance of a film increases, or conversely, the reflectivity and the corrosion resistance of a film formed through sputtering decrease, which is not preferable. Therefore, since the composition of a film depends on the composition of the target, the content of In contained in the silver-alloy sputtering target is set to be in a range of 0.1 mass% to 1.5 mass%. The content of In is more preferably in a range of 0.2 mass% to 1.0 mass%.

[0025]

A silver-alloy sputtering target for conductive-film formation of a second embodiment is made of a silver alloy having a component composition including In and Sn at a total content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag and inevitable impurities. The average grain diameter of crystal grains in the alloy is in a

range of 30 μm or more to less than 150 μm , and the dispersion of the grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.

In the present embodiment, similarly to In, Sn is solid-solubilized in Ag so as to suppress the crystal grain growth in the target; and therefore, Sn is effective for refining crystal grains. Since In and Sn improve the hardness of the target, In and Sn suppress warpage during machining. In and Sn improve the corrosion resistance and the heat resistance of a film formed through sputtering. In the case where the total content of In and Sn is less than 0.1 mass%, the above-described effects cannot be obtained, and in the case where the total content of In and Sn exceeds 1.5 mass%, the reflectivity or the electric resistance of the film decreases.

[0026]

A silver-alloy sputtering target for conductive-film formation of a third embodiment is made of a silver alloy having a component composition including: In at a content in a range of 0.1 mass% to 1.5 mass%; and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag and inevitable impurities. The average grain diameter of crystal grains in the alloy is in a range of 30 μm or more to less than 150 μm , and the dispersion of the grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.

In addition, a silver-alloy sputtering target for conductive-film formation of a fourth embodiment is made of a silver alloy having a component composition including: In and Sn at a total content in a range of 0.1 mass% to 1.5 mass%; and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag and inevitable impurities. The average grain diameter of crystal grains in the alloy is in a range of 30 μm or more to less than 150 μm , and the dispersion of the grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.

[0027]

In the third and fourth embodiments, Sb and Ga are solid-solubilized in Ag so as to further suppress crystal grain growth. Sb and Ga further improve the corrosion resistance and the heat resistance of a film formed through sputtering. Particularly, Ga improves the resistance against chloridation of the film. In the case where a film formed through sputtering is used as a lead-out wiring film in a touch panel, the wiring film is required to have a resistance against a chlorine component contained in sweat from a human body because the touch panel is operated by the touch of a finger. A film having excellent resistance against chloridation can be formed by adding Ga.

In the case where the total content of Sb and Ga is less than 0.1 mass%, the above-described effects cannot be obtained. In the case where the total content of Sb and Ga exceeds 2.5 mass%, not only does the reflectivity or the electric resistance of the film decrease, but also cracking tends to occur during hot rolling.

[0028]

In the above-described embodiments having the respective compositions, the average grain diameter of silver alloy crystal grains in the silver-alloy sputtering target is in a range of 30 μm or more to less than 150 μm , and preferably in a range of 30 μm or more to less than 120 μm . Regarding the average grain diameter of the silver alloy crystal grains, an average grain diameter of less than 30 μm is not realistic, and this causes an increase in the production cost. In addition, it is difficult to produce uniform crystal grains, and the dispersion of the grain diameters increases. Therefore, abnormal discharge is likely to occur during high-power sputtering, and splash is caused. On the other hand, in the case where the average grain diameter is 150 μm or more, as the target is consumed by a sputter, the unevenness of the sputter surface becomes large due to the

difference in the sputter rate caused by the difference in crystal orientation among individual crystal grains. Therefore, abnormal discharge is likely to occur during high-power sputtering, and splash is likely to be caused. In the case where the average crystal grain diameter is set to be in a range of less than 120 μm , it is possible to further suppress arc discharging and splash.

[0029]

Here, the average grain diameter of the silver alloy crystal grains is measured as described below.

Rectangular parallelepiped specimens approximately 10 mm on a side are sampled from 16 places at even intervals in a surface to be sputtered of the target. Specifically, the target is divided into 16 (4×4) segments, and a specimen is sampled from the central portion of each segment. Meanwhile, in the embodiment, since a surface to be sputtered having dimensions of 500 mm×500 mm or more, that is, a large target which includes the target surface having an area of 0.25 m² or more is taken into account, a method for sampling a specimen from a rectangular target normally used as a large target will be described. However, needless to say, the invention also exhibits an effect of suppressing the occurrence of splash in a round target. In this case, the surface to be sputtered of the target is evenly divided into 16 segments, and specimens are sampled according to the method for sampling a specimen from a large rectangular target.

Next, the surfaces to be sputtered of the respective specimens are polished. At this time, the polishing is carried out using water proof paper of #180 to #4000, and then buffing is carried out using abrasive grains having sizes of 3 μm to 1 μm .

Furthermore, the surfaces are etched until grain boundaries become visible using an optical microscope. Here, a mixed solution of hydrogen peroxide solution and

ammonia water is used as an etching solution, the surfaces are immersed for one second to two seconds at room temperature; and thereby, the grain boundaries are made to appear.

Next, photographs of the respective specimens are taken using the optical microscope at a magnification of 60 times to 120 times. Regarding the magnification of the photograph,

5 a magnification facilitating the counting of crystal grains is selected.

On each photograph, a total of four 60 mm-long lines are drawn horizontally and perpendicularly in a grid shape (like # sign) at intervals of 20 mm, and the number of crystal grains intersected by the respective straight lines is counted. Meanwhile, a crystal grain on the edge of the line is counted as 0.5 grains. An average section length (L , μm)

10 is obtained using $L = 60000/(M \cdot N)$ (here, M represents the actual magnification, and N represents the average value of the number of intersected crystal grains).

Next, the average grain diameter (d , μm) of the specimen is computed from the obtained average section length (L , μm) using $d = (3/2) \cdot L$.

The average value of the average grain diameters of the specimens sampled from
15 16 places as described above is considered as the average grain diameter of silver alloy crystal grains in the target.

[0030]

In the case where the dispersion of the grain diameter of the silver alloy crystal grains is in a range of 20% or less of the average grain diameter of the silver alloy crystal
20 grains, splash during sputtering can be more reliably suppressed. Here, the dispersion of the grain diameter is computed as described below. Among the 16 average grain diameters obtained from 16 places, the average grain diameter having the greatest deviation in the absolute value ($|[(\text{the average grain diameter at a certain place}) - (\text{the average value of the average grain diameters at 16 places})]|$) from the average value of

the average grain diameters is specified. Next, the dispersion of the grain diameter is computed using the specified average grain diameter (specific average grain diameter) and the following formula.

$$\{ | [(\text{the specific average grain diameter}) - (\text{the average value of the average grain diameters at 16 places})] | / (\text{the average value of the average grain diameters at 16 places}) \} \times 100 (\%)$$

[0031]

Next, a method for producing a silver-alloy sputtering target for conductive-film formation of the embodiment will be described.

10 In a method for producing a silver-alloy sputtering target for conductive-film formation of a first embodiment, Ag with a purity of 99.99 mass% or more and In with a purity of 99.9 mass% or more are used as raw materials.

First, Ag is melted in a high vacuum or inert gas atmosphere, and a predetermined content of In is added to the obtained molten silver. After that, the mixture is melted in a vacuum or inert gas atmosphere so as to manufacture a cast ingot of a silver alloy including In at a content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag and inevitable impurities.

Here, the melting of Ag and the addition of In are preferably carried out as described below. The atmosphere is once evacuated, and then, a vacuum atmosphere is replaced with argon gas, and Ag is melted in this atmosphere. Next, In is added to molten Ag in the argon atmosphere after the melting of Ag. Thereby, the composition ratio between Ag and In becomes stable.

[0032]

In a method for producing a silver-alloy sputtering target for conductive-film formation of a second embodiment, Ag with a purity of 99.99 mass% or more, In and Sn

having a purity of 99.9 mass% or more respectively are used as raw materials. In is melted in molten Ag, and Sn is added so that the total content of In and Sn becomes in a range of 0.1 mass% to 1.5 mass%. In this case as well, Ag is melted in a high vacuum or inert gas atmosphere, In and Sn are added at predetermined contents to the obtained
5 molten silver, and then the mixture is melted in a vacuum or inert gas atmosphere.

[0033]

In a method for producing a silver-alloy sputtering target for conductive-film formation of a third embodiment, Ag with a purity of 99.99 mass% or more and In, Sb, and Ga having a purity of 99.9 mass% or more respectively are used as raw materials. In
10 at a content in a range of 0.1 mass% to 1.5 mass% and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 3.0 mass% are added to molten Ag. In this case as well, Ag is melted in a high vacuum or inert gas atmosphere, In, Sb and Ga are added at predetermined contents to the obtained molten silver, and then the mixture is melted in a vacuum or inert gas atmosphere.

15 [0034]

In a method for producing a silver-alloy sputtering target for conductive-film formation of a fourth embodiment, Ag with a purity of 99.99 mass% or more and In, Sn, Sb, and Ga having a purity of 99.9 mass% or more respectively are used as raw materials. In and Sn at a total content in a range of 0.1 mass% to 1.5 mass% are added to molten Ag,
20 and either one or both of Sb and Ga are further added at a total content in a range of 0.1 mass% to 3.0 mass%. In this case as well, Ag is melted in a high vacuum or inert gas atmosphere, In, Sn, Sb, and Ga are added at predetermined contents to the obtained molten silver, and then the mixture is melted in a vacuum or inert gas atmosphere.

[0035]

25 In addition, the above-described melting and casting are desirably carried out in a

vacuum or in an inert gas-substituted atmosphere, but it is also possible to use a melting furnace in air. In the case where a melting furnace in air is used, the melting and casting are carried out while blowing an inert gas to the surface of the molten silver alloy or covering the surface of the molten silver alloy with a carbon-based solid sealing material
 5 such as coal and the like. Thereby, it is possible to reduce the content of oxygen and non-metallic inclusions in the ingot.

The melting furnace is preferably an induction heating furnace for homogenizing the components.

In addition, it is efficient and desirable to carry out casting using a square casting
 10 mold to obtain a rectangular parallelepiped ingot, but it is also possible to cast a cylindrical ingot in a round casting mold and to work the cylindrical ingot to obtain a substantially rectangular parallelepiped ingot.

[0036]

The obtained rectangular parallelepiped ingot is heated and hot-rolled to a
 15 predetermined thickness, and then the ingot is quenched.

In this case, the conditions of finish hot rolling in the final phase of hot rolling are important, and by setting the finish hot rolling conditions appropriately, it is possible to produce a silver alloy sheet having fine homogeneous crystal grains.

Specifically, in the finish hot rolling, the rolling reduction rate per pass is set to be
 20 in a range of 20% to 50%, the strain rate is set to be in a range of 3 /sec to 15 /sec, and the rolling temperature after each rolling pass is set to be in a range of 400°C to 650°C. One or more passes of this finish hot rolling are carried out. The total rolling reduction rate throughout the hot rolling is set to be, for example, in a range of 70% or more.

Here, the finish hot rolling refers to a rolling pass having a strong effect on the
 25 crystal grain diameter of a sheet material after rolling, includes the final rolling pass, and

may be considered as three consecutive passes from the final rolling pass as necessary. After the final rolling, it is also possible to carry out additional rolling at a rolling reduction rate of 7% or less in the above-described rolling temperature range so as to adjust the sheet thickness.

5 In addition, the strain rate ε (sec^{-1}) is considered to be expressed by the following formula.

[0037]

[Formula 1]

$$\varepsilon = \frac{2\pi n}{60\sqrt{r'}} \cdot \sqrt{\frac{R}{H_0}} \cdot \ln\left(\frac{1}{1-r'}\right)$$

10 [0038]

In the above-described formula, H_0 represents the sheet thickness (mm) on the inlet side of a rolling roll, n represents the rotation rate (rpm) of the rolling roll, R represents the radius (mm) of the rolling roll, r represents the rolling reduction rate (%), and $r' = r/100$.

15 In the case where the rolling reduction rate per pass is set to be in a range of 20% to 50% and the strain rate is set to be in a range of 3 /sec to 15 /sec, the ingot is strongly worked using a large energy at a relatively low temperature. Thereby, the mixture of coarse crystal grains is prevented, and it is possible to generate fine homogeneous crystal grains as a whole through dynamic recrystallization. In the case where the rolling
20 reduction rate per pass is less than 20%, the crystal grains fail to be sufficiently refined. When an attempt is made to obtain a rolling reduction rate of more than 50%, the load of a rolling machine becomes excessive, which is not realistic. In addition, in the case where

the strain rate is less than 3 /sec, crystal grains fail to be sufficiently refined, and mixed grains of fine grains and coarse grains tend to be generated. When an attempt is made to obtain a strain rate of more than 15 /sec, the load of the rolling machine becomes excessive, which is not realistic.

5 [0039]

The rolling temperature after each pass is set to be in a range of 400°C to 650°C that is a low temperature for hot rolling. Thereby, the coarsening of the crystal grains is suppressed. In the case where a rolling temperature is lower than 400°C, dynamic recrystallization becomes insufficient, and the dispersion of the crystal grain diameter
10 tends to become greater. In the case where the rolling temperature exceeds 650°C, crystal grains grow, and the average crystal grain diameter becomes greater than 150 μm .

One pass of the final finish hot rolling is carried or multiple passes of the final finish hot rolling are carried as necessary.

The more preferable conditions of the finish hot rolling are as follows: a rolling
15 reduction rate per pass is in a range of 25% to 50%; a strain rate is in a range of 5 /sec to 15 /sec; and a rolling temperature after the pass is in a range of 500°C to 600°C, and it is preferable to carry out three passes or more of the finish hot rolling.

Meanwhile, the rolling start temperature may not be in a range of 400°C to 650°C, and the rolling start temperature and the pass schedule are set so that the temperature after
20 each pass becomes in a range of 400°C to 650°C in the finish hot rolling in the final phase.

[0040]

In addition, after the above-described hot rolling step, quenching is carried out at a cooling rate in a range of 200 °C/min to 1000 °C/min from a temperature in a range of 400°C to 650°C to, for example, a temperature of 200°C or lower. The quenching

suppresses the growth of crystal grains; and thereby, a rolled sheet including fine crystal grains can be obtained. In the case where the cooling rate is less than 200 °C/min, the effect of suppressing the growth of crystal grain is not sufficient. Even in the case where the cooling rate exceeds 1000 °C/min, there is no contribution to additional refinement.

- 5 As a method of the quenching, the sheet may be showered using water for approximately one minute.

[0041]

- The rolled sheet obtained in the above-described manner is straightened using a straightening press, a roller leveler and the like, and then the rolled steel is subjected to
10 machining such as milling or electrical discharge machining so as to be finished into desired dimensions. The arithmetic average roughness (Ra) of the sputter surface of the finally-obtained sputtering target is preferably in a range of 0.2 μm to 2 μm.

[0042]

- The silver-alloy sputtering target for conductive-film formation of the
15 embodiment obtained in the above-described manner is capable of suppressing abnormal discharge and is also capable of suppressing the occurrence of splash even when a large power is supplied in the middle of sputtering. When this target is sputtered, a conductive film having a high reflectivity and excellent durability can be obtained. In addition, when sputtering is carried out using the silver-alloy sputtering target for conductive-film
20 formation, it is possible to obtain a conductive film having favorable corrosion resistance and heat resistance and furthermore, having a low electric resistance. Particularly, the silver-alloy sputtering target for conductive-film formation is effective in the case of a large target having a target size of width: 500 mm, length: 500 mm and thickness: 6 mm or more.

EXAMPLES

[0043]

(Example 1)

Ag with a purity of 99.99 mass% or more and In with a purity of 99.9 mass% or
5 more as an additive raw material were prepared and loaded into a high-frequency
induction melting furnace installed with a graphite crucible. The total mass during
melting was set to approximately 1100 kg.

In the melting, Ag was first melted, and, after Ag was melted down, the additive
raw material was added so as to obtain a target composition described in Table 1. The
10 molten alloy was sufficiently stirred using the stirring effect of induction heating, and then
the molten alloy was cast in a casting mold made of cast iron.

Shrink holes in the ingot obtained through the casting were removed, and the
surface that had been in contact with the casting mold was ground and removed; and
thereby, a rectangular parallelepiped ingot was obtained which included a sound part
15 having approximate dimensions of 640×640×180 (mm).

[0044]

The ingot was heated to 770°C, and the ingot was repeatedly rolled in a single
direction so as to be stretched to have a length of 640 mm to 1700 mm. The stretched
ingot was rotated by 90 degrees, and then rolling was repeatedly carried out in another 640
20 mm-long direction; and thereby, a sheet material was manufactured which had
approximate dimensions of 1700×2100×20 (mm).

In the hot rolling, a total of 12 passes were repeated. Among those passes, the
conditions for the three consecutive passes from the final pass (the strain rate per pass, the
rolling reduction rate and the sheet material temperature after the pass) were set as
25 described in Table 1. The total rolling reduction rate of the entire hot rolling was 89%.

After the end of the hot rolling, the sheet material after the rolling was cooled under the conditions described in Table 3.

After the cooling, the sheet material was passed through a roller leveler so as to straighten strains generated by the quenching, and the sheet material was machined to have dimensions of 1600×2000×15 (mm); and thereby, a target was manufactured.

[0045]

(Examples 2 to 10 and Comparative Examples 1 to 10)

The heating temperature of an ingot before the hot rolling was changed in a range of 510°C to 880°C, the sheet thickness after the final rolling was changed in a range of 9.0 mm to 24.2 mm, the total number of the passes was changed in a range of 10 times to 14 times, the total rolling reduction rate was changed in a range of 87% to 95% in the same manner as in Example 1. In addition, a target was manufactured with the target composition described in Table 3 under the condition for the three consecutive passes from the final pass described in Table 1 and the condition of the cooling rate after the hot rolling described in Table 3. In Table 3, a target of which the cooling rate was described in the column of “Cooling rate” indicates a target which was cooled through water showering, and a target with “no water cooling” described in the column of “Cooling rate” indicates a target that was simply cooled in the air. However, the thickness of the target after machining was set to be in a range of 6 mm to 21 mm.

[0046]

(Examples 11 to 13 and Comparative Example 11)

Raw materials were melted and cast in the same manner as in Example 1; and thereby, an ingot was manufactured which had approximate dimensions of 640×640×60 (mm). The ingot was heated to 700°C, and then the ingot was hot-rolled; and thereby, a

sheet material was manufactured which had approximate dimensions of 1200×1300×16 (mm).

In the hot rolling, a total of six passes were repeated. Among those passes, the conditions for the three consecutive passes from the final pass (the strain rate per pass, the rolling reduction rate and the sheet material temperature after the pass) were set as described in Table 2. The total rolling reduction rate of the entire hot rolling was 73%.

After the end of the hot rolling, the sheet material after the rolling was cooled under the conditions described in Table 3.

After the cooling, the sheet material was passed through a roller leveler so as to straighten strains generated by the quenching, and the sheet material was machined to have dimensions of 1000×1200×12 (mm); and thereby, a target was manufactured.

[0047]

(Example 14 and Comparative Example 12)

Ag with a purity of 99.99 mass% or more and In and Sn having purities of 99.9 mass% or more respectively as additive raw materials were prepared. In the high-frequency induction melting furnace installed with a graphite crucible, Ag was first melted, and, after Ag was melted down, the additive raw materials were added so as to obtain a target composition described in Table 3. The molten alloy was sufficiently stirred using the stirring effect of induction heating, and then the molten alloy was cast in a cast iron casting mold.

[0048]

(Examples 15 to 22 and Comparative Example 13 to 15)

Ag with a purity of 99.99 mass% or more and In, Sb and Ga having purities of 99.9 mass% or more respectively as additive raw materials were prepared. In the high-frequency induction melting furnace installed with a graphite crucible, Ag was first

melted, and, after Ag was melted down, the additive raw materials were added so as to obtain a target composition described in Table 3. The molten alloy was sufficiently stirred using the stirring effect of induction heating, and then the molten alloy was cast in a cast iron casting mold.

5 [0049]

(Example 23)

Ag with a purity of 99.99 mass% or more and In, Sn, Sb and Ga having purities of 99.9 mass% or more respectively as additive raw materials were prepared. In the high-frequency induction melting furnace installed with a graphite crucible, Ag was first
10 melted, and, after Ag was melted down, the additive raw materials were added so as to obtain a target composition described in Table 3. The molten alloy was sufficiently stirred using the stirring effect of induction heating, and then the molten alloy was cast in a cast iron casting mold.

[0050]

15 In Examples 14 to 23 and Comparative Examples 12 to 15, after casting, an ingot having approximate dimensions of 640×640×60 (mm) was manufactured using an ingot obtained through casting in the same manner as in Examples 11 to 13 and Comparative Example 11. Next, the ingot was heated up to 700°C, and then the ingot was hot-rolled in the same manner as described above; and thereby, a sheet material was manufactured
20 which had approximate dimensions of 1200×1300×16 (mm).

In the hot rolling, a total of six passes were repeated. Among those passes, the conditions for the three consecutive passes from the final pass (the strain rate per pass, the rolling reduction rate and the sheet material temperature after the pass) were set as described in Table 2. The total rolling reduction rate of the entire hot rolling was 73%.

25 Then, the sheet material was cooled under the conditions described in Table 3. Next, the

sheet material was passed through a roller leveler so as to straighten strains generated by the quenching, and the sheet material was machined to have dimensions of 1000×1200×12 (mm); and thereby, a target was manufactured.

[0051]

Table 1

	Finish hot rolling conditions								
	Third pass from final rolling pass			Second pass from final rolling pass			Final rolling pass		
	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)
Comparative Example 1	2.1	19	510	2.5	20	490	2.8	20	470
Example 1	5.0	26	570	5.7	25	540	8.5	36	510
Example 2	2.2	21	530	2.6	23	500	3.0	23	480
Example 3	2.8	17	550	3.4	21	520	4.2	24	500
Example 4	3.7	23	490	4.3	24	470	5.1	25	450
Example 5	3.9	10	610	8.6	33	580	14.8	50	550
Comparative Example 2	5.5	30	380	6.6	30	360	8.0	31	350
Example 6	4.4	23	440	5.6	27	420	7.2	31	400
Example 7	5.0	26	550	6.9	33	530	8.3	32	510
Example 8	5.2	28	600	6.9	33	580	8.4	33	550

Table 1 (Continued)

	Finish hot rolling conditions								
	Third pass from final rolling pass			Second pass from final rolling pass			Final rolling pass		
	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)
Example 9	5.3	25	650	6.6	28	630	9.3	36	600
Comparative Example 3	2.9	21	730	3.4	23	700	3.8	23	670
Comparative Example 4	3.2	17	540	3.6	18	520	4.0	18	500
Example 10	5.1	20	540	5.8	21	520	6.5	21	500
Comparative Example 5	4.9	22	650	6.6	29	630	7.8	29	600
Comparative Example 6	4.9	26	630	6.4	30	610	8.2	33	580
Comparative Example 7	2.0	17	500	2.2	17	470	2.4	16	450
Comparative Example 8	4.6	15	360	5.2	17	350	6.0	18	330
Comparative Example 9	2.0	24	720	2.3	24	690	2.7	25	660
Comparative Example 10	5.2	24	630	6.4	27	610	7.6	28	580

[0052]

Table 2

	Finish hot rolling conditions								
	Third pass from final rolling pass			Second pass from final rolling pass			Final rolling pass		
	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)	Strain rate (/sec)	Rolling reduction rate (%)	Temperature after pass (°C)
Example 11	5.5	24	590	7.0	28	550	8.8	30	520
Example 12	5.5	24	580	7.0	28	540	8.8	30	510
Example 13	5.5	24	560	7.0	28	530	8.8	30	500
Comparative Example 11	5.5	24	560	7.0	28	530	8.8	30	500
Example 14	5.5	24	570	7.0	28	540	8.8	30	500
Comparative Example 12	5.5	24	590	7.0	28	550	8.8	30	520
Example 15	5.5	24	560	7.0	28	530	8.8	30	500
Example 16	5.5	24	590	7.0	28	560	8.8	30	530
Example 17	5.5	24	600	7.0	28	560	8.8	30	520
Example 18	5.5	24	550	7.0	28	530	8.8	30	500
Comparative Example 13	5.5	24	550	7.0	28	520	8.8	30	490
Example 19	5.5	24	560	7.0	28	530	8.8	30	500
Example 20	5.5	24	580	7.0	28	550	8.8	30	520
Example 21	5.5	24	580	7.0	28	540	8.8	30	510
Comparative Example 14	5.5	24	580	7.0	28	550	8.8	30	510
Example 22	5.5	24	570	7.0	28	540	8.8	30	500
Comparative Example 15	5.5	24	580	7.0	28	540	8.8	30	510
Example 23	5.5	24	560	7.0	28	530	8.8	30	500

[0053]

Table 3

	Cooling rate (°C/min)	Target composition (mass%)				
		In	Sn	Sb	Ga	Ag
Comparative Example 1	300	0.7	0	0	0	Remainder
Example 1	300	0.7	0	0	0	Remainder
Example 2	300	0.7	0	0	0	Remainder
Example 3	350	0.7	0	0	0	Remainder
Example 4	300	0.7	0	0	0	Remainder
Example 5	350	0.7	0	0	0	Remainder
Comparative Example 2	300	0.5	0	0	0	Remainder
Example 6	200	0.5	0	0	0	Remainder
Example 7	350	0.5	0	0	0	Remainder
Example 8	400	0.5	0	0	0	Remainder
Example 9	400	0.5	0	0	0	Remainder
Comparative Example 3	400	0.5	0	0	0	Remainder
Comparative Example 4	300	0.6	0	0	0	Remainder
Example 10	300	0.6	0	0	0	Remainder
Comparative Example 5	No water cooling	0.6	0	0	0	Remainder
Comparative Example 6	100	0.6	0	0	0	Remainder
Comparative Example 7	300	0.6	0	0	0	Remainder
Comparative Example 8	150	0.6	0	0	0	Remainder
Comparative Example 9	400	0.6	0	0	0	Remainder
Comparative Example 10	350	0	0	0	0	Remainder
Example 11	350	0.2	0	0	0	Remainder
Example 12	350	1	0	0	0	Remainder
Example 13	350	1.5	0	0	0	Remainder
Comparative Example 11	350	1.7	0	0	0	Remainder
Example 14	350	0.5	0.5	0	0	Remainder
Comparative Example 12	350	1	0.6	0	0	Remainder
Example 15	350	0.5	0	0.1	0	Remainder
Example 16	350	0.5	0	0.5	0	Remainder
Example 17	350	0.5	0	1.5	0	Remainder
Example 18	350	0.5	0	2.4	0	Remainder
Comparative Example 13	350	0.5	0	2.7	0	Remainder
Example 19	350	0.4	0	0	0.1	Remainder
Example 20	350	0.4	0	0	1.5	Remainder
Example 21	350	0.4	0	0	2.4	Remainder
Comparative Example 14	350	0.4	0	0	2.7	Remainder
Example 22	350	0.5	0	1.2	1.2	Remainder
Comparative Example 15	350	0.5	0	1.4	1.3	Remainder
Example 23	350	0.4	0.4	0.6	0.6	Remainder

[0054]

With regard to the obtained targets, warpage after the machining, the average grain diameter, and the dispersion of the average grain diameter were measured. In addition, the target was installed in a sputtering device, and the number of times of occurring abnormal discharge during sputtering was measured. Furthermore, for conductive films obtained through sputtering, the surface roughness, the reflectivity, the resistance against chloridation and the specific resistance were measured.

(1) Warpage after the machining

The warpage amount per length of 1 m was measured in the silver-alloy sputtering target after the machining, and the result is described in Table 4.

(2) Average grain diameter and dispersion of the average grain diameter

The grain diameters of silver alloy crystal grains were measured using the method described in the EMBODIMENTS OF THE INVENTION section. In detail, specimens were sampled from 16 places at even intervals on the target produced in the above-described manner, and the average grain diameter in a surface visible to the sputtered surface of each specimen was measured. Then, the average grain diameter of silver alloy crystal grains that was an average value of the average grain diameters of the respective specimens and the dispersion of the average grain diameter of the silver alloy crystal grains were computed.

[0055]

(3) Number of times of occurring abnormal discharge during sputtering

A round sheet having a diameter of 152.4 mm and a thickness of 6 mm was cut out from an arbitrary section of the target produced as described above, and the round sheet was soldered to a copper backing plate. The soldered target was used as a target for splash evaluation during sputtering, and the number of times of occurring abnormal

discharge during sputtering was measured.

In this case, the soldered target was installed in an ordinary magnetron sputtering apparatus, and the air was exhausted until the pressure reached 1×10^{-4} Pa. Next,

sputtering was carried out under conditions where an Ar gas pressure was 0.5 Pa, a

5 supplied power of DC was 1000 W and a distance between the target and the substrate was 60 mm. The number of times of occurring abnormal discharge occurring for 30 minutes

from the beginning of the use of the target was measured. In addition, the target was

consumed by continuously carrying out sputtering for 20 hours through the repetition of four-hour sputtering without a substrate and the exchange of an adhesion shield sheet.

10 After that, sputtering was further carried out, and the number of times of occurring abnormal discharge occurring for 30 minutes after the consumption (20-hour sputtering) was measured. The number of times of occurring the abnormal discharge was measured using an arc counting function of a DC power generator (Model No. RPDG-50A) manufactured by MKS Instruments.

15 [0056]

(4) Evaluation of the basic characteristics as a conductive film

(4-1) Surface roughness of the film

Sputtering was carried out under the same conditions as described above using the target for evaluation, and a silver alloy film having a film thickness of 100 nm was
20 formed on a 20×20 (mm) glass substrate. Furthermore, in order to evaluate the heat resistance, the silver alloy film was subjected to a heat treatment at 250°C for 10 minutes. After that, the average surface roughness (Ra) of the silver alloy film was measured using an atomic force microscope.

(4-2) Reflectivity

25 A silver alloy film was formed on a 30×30 (mm) glass substrate in the same

manner as described above. In addition, the absolute reflectivity of the silver alloy film at a wavelength of 550 nm was measured using a spectrophotometer.

Furthermore, in order to evaluate the corrosion resistance, the silver alloy film was held in a constant-temperature and high-humidity chamber having a temperature of 80°C and a humidity of 85% for 100 hours. After that, the absolute reflectivity of the silver alloy film at a wavelength of 550 nm was measured using a spectrophotometer.

(4-3) Resistance against chloridation

In order to check the effect of the addition of Ga, a silver alloy film was formed in the same manner as described above using a Ga-added target (Examples 19 to 23 and Comparative Examples 14 and 15). Next, an aqueous solution containing 5% by weight of NaCl was sprayed to the film surface of the silver alloy film. The aqueous solution was sprayed in a direction parallel to the film surface from a location 20 cm high from the film surface and 10 cm distant from the edge of the substrate, and the NaCl aqueous solution sprayed on the film was left to fall freely as much as possible and be attached to the film. The aqueous solution was sprayed five times every one minute, and then the film was washed using pure water three times. Moisture was blown off by spraying dried air, and the film was dried.

The silver alloy film surface after the above-described spraying of saline water was visually observed, and the state of the surface was evaluated. Regarding the evaluation criteria of the resistance against chloridation, a film surface which did not become white-turbid or spotted or became only partially white-turbid or spotted was evaluated to be “favorable”, and a film surface which became fully white-turbid or spotted was evaluated to be “poor”. The surface state was evaluated into two levels using the above-described criteria. For the target to which Ga had not been added, no evaluation was carried out, and thus “-” was marked in the table.

(4-4) Specific resistance of the film

The specific resistance of the silver alloy film formed in the same manner as described above was measured.

The respective evaluation results are described in Tables 4 to 6.

5 [0057]

Table 4

	Average grain diameter (μm)	Dispersion of grain diameter (%)	Warpage after machining (mm)	Number of times of abnormal discharge (times)	
				At the beginning of use	After consumption
Comparative Example 1	180	23	0.6	5	12
Example 1	80	13	0.5	0	0
Example 2	140	19	0.5	3	6
Example 3	130	18	0.6	1	3
Example 4	90	15	0.7	0	1
Example 5	60	14	0.4	0	0
Comparative Example 2	70	35	0.7	10	38
Example 6	90	18	0.6	1	2
Example 7	100	14	0.6	1	1
Example 8	110	13	0.5	0	0
Example 9	140	13	0.5	1	4
Comparative Example 3	170	11	0.4	2	12
Comparative Example 4	190	25	0.7	5	15
Example 10	140	18	0.7	3	4
Comparative Example 5	190	14	0.6	2	12
Comparative Example 6	170	15	0.5	3	12
Comparative Example 7	210	29	0.6	13	46
Comparative Example 8	170	44	0.5	15	61
Comparative Example 9	250	14	0.7	2	23

Table 4 (Continued)

	Average grain diameter (μm)	Dispersion of grain diameter (%)	Warpage after machining (mm)	Number of times of abnormal discharge (times)	
				At the beginning of use	After consumption
Comparative Example 10	160	15	2.8	2	13
Example 11	140	18	1.2	3	4
Example 12	120	15	0.6	0	1
Example 13	90	14	0.6	1	0
Comparative Example 11	80	14	0.6	0	0
Example 14	90	14	0.6	0	1
Comparative Example 12	80	15	0.5	1	0
Example 15	60	16	0.5	1	0
Example 16	50	16	0.6	0	0
Example 17	45	18	0.6	1	1
Example 18	35	18	0.8	1	1
Comparative Example 13	35	18	Cracked	1	1
Example 19	60	17	0.6	0	0
Example 20	50	18	0.5	0	0
Example 21	45	17	0.8	0	0
Comparative Example 14	35	17	Cracked	0	1
Example 22	40	18	0.7	0	1
Comparative Example 15	40	18	Cracked	1	0
Example 23	50	18	0.6	0	0

[0058]

Table 5

	Absolute reflectivity of film (%)	Specific resistance of film ($\mu\Omega\cdot\text{cm}$)	Absolute reflectivity after holding of film at constant temperature and constant humidity (%)	Evaluation of resistance against chloridation of film	Surface roughness after heat treatment of film Ra (nm)
Comparative Example 1	96.6	4.4	96.4	-	0.8
Example 1	96.6	4.3	96.5	-	0.9
Example 2	96.7	4.3	96.5	-	0.8
Example 3	96.5	4.4	96.4	-	0.9
Example 4	96.7	4.4	96.4	-	0.9
Example 5	96.5	4.3	96.4	-	0.8
Comparative Example 2	96.9	4.1	96.7	-	0.9
Example 6	96.8	4.1	96.7	-	0.9
Example 7	96.8	4.2	96.6	-	0.8
Example 8	96.7	4.1	96.6	-	0.9
Example 9	96.8	4.1	96.6	-	0.9
Comparative Example 3	96.8	4.2	96.5		0.9
Comparative Example 4	96.6	4.3	96.4	-	0.8
Example 10	96.7	4.2	96.5	-	0.9
Comparative Example 5	96.6	4.3	96.4	-	0.8
Comparative Example 6	96.7	4.2	96.4	-	0.8
Comparative Example 7	96.6	4.3	96.3	-	0.9
Comparative Example 8	96.7	4.3	96.5	-	0.8
Comparative Example 9	96.6	4.2	96.4	-	0.9
Comparative Example 10	97.6	3.2	89.7	-	2.3

[0059]

Table 6

	Absolute reflectivity of film (%)	Specific resistance of film ($\mu\Omega\cdot\text{cm}$)	Absolute reflectivity after holding of film at constant temperature and constant humidity (%)	Evaluation of resistance against chloridation of film	Surface roughness after heat treatment of film Ra (nm)
Example 11	97.1	3.6	96.2	-	1.2
Example 12	95.9	4.6	95.5	-	0.7
Example 13	95.5	5.9	95.0	-	0.7
Comparative Example 11	92.9	7.5	86.5	-	0.7
Example 14	96.2	4.6	95.9	-	0.8
Comparative Example 12	91.3	7.5	88.3	-	0.7
Example 15	96.8	4.4	96.6	-	0.7
Example 16	96.6	5.2	94.4	-	0.6
Example 17	95.0	6.4	94.0	-	0.5
Example 18	94.5	6.9	93.5	-	0.5
Comparative Example 13	92.1	8.8	88.1	-	0.5
Example 19	96.7	4.4	96.4	Favorable	0.7
Example 20	94.9	6.8	91.8	Favorable	0.6
Example 21	93.8	7.2	90.5	Favorable	0.6
Comparative Example 14	91.3	10.2	83.9	Favorable	0.5
Example 22	93.9	7.1	91.0	Favorable	0.6
Comparative Example 15	91.1	10.5	84.3	Favorable	0.5
Example 23	94.8	6.7	93.5	Favorable	0.6

[0060]

In the target materials of the examples, the average grain diameters of the silver alloy crystal grains were in a range of 30 μm or more to less than 150 μm , and the dispersions of the grain diameters of the silver alloy crystal grains were in a range of 20% or less of the average grain diameters of the silver alloy crystal grains. The warpage after machining was small, and the number of times of occurring abnormal discharge during sputtering was also small not only at the beginning of the use of the target but also after the consumption of the target. Particularly, in the case where the average crystal grain diameter was in a range of 120 μm or less and the dispersion of the grain diameter was in a range of 20% or less, the number of times of occurring abnormal discharge was one or less which was small in all the examples except for Example 6 in which the number was two after the consumption. In addition, the targets to which Sb and Ga were added tended to have a small average crystal grain diameter and had a small number of times of occurring abnormal discharge that was one or less. However, in the target in which the addition amount of Sb and Ga was excessive (greater than 2.5 mass% in total), cracking occurred during the machining.

In addition, in the conductive films obtained using the target material of the examples, the reflectivity and the specific resistance were excellent, and the surface roughness Ra was also as small as 2 μm or less.

In addition, it was found that the conductive films obtained using the Ga-added target also had excellent resistance against chloridation, and was effective for a conductive film in a touch panel or the like.

[0061]

Meanwhile, the invention is not limited to the above-described embodiment, and a variety of modifications can be added within the scope of the features of the invention.

Industrial Applicability

[0062]

When the target of the embodiment is sputtered, the occurrence of arc discharging
5 and splash is suppressed. In addition, a conductive film obtained by sputtering the target
of the embodiment has an excellent reflectivity and excellent resistivity, and also has a
small surface roughness. Therefore, the target of the embodiment is preferably
applicable as a target for forming a conductive film such as a reflective electrode layer in
an organic EL element or a wiring film in a touch panel.

CLAIMS

1. A silver-alloy sputtering target for conductive-film formation having a component composition comprising In at a content in a range of 0.1 mass% to 1.5 mass% with a
5 remainder of Ag and inevitable impurities,
wherein an average grain diameter of crystal grains in an alloy is in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.
- 10 2. A silver-alloy sputtering target for conductive-film formation having a component composition comprising In and Sn at a total content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag and inevitable impurities,
wherein an average grain diameter of crystal grains in an alloy is in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of the crystal grains
15 is in a range of 20% or less of the average grain diameter.
3. A silver-alloy sputtering target for conductive-film formation having a component composition comprising: In at a content in a range of 0.1 mass% to 1.5 mass%; and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a
20 remainder of Ag and inevitable impurities,
wherein an average grain diameter of crystal grains in an alloy is in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.
- 25 4. A silver-alloy sputtering target for conductive-film formation having a component

composition comprising: In and Sn at a total content in a range of 0.1 mass% to 1.5 mass%; and either one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag and inevitable impurities,

wherein an average grain diameter of crystal grains in an alloy is in a range of 30 μm or more to less than 150 μm , and a dispersion of grain diameters of the crystal grains is in a range of 20% or less of the average grain diameter.

5. The silver-alloy sputtering target for conductive-film formation according to any one of Claims 1 to 4,

wherein the average grain diameter of the crystal grains is in a range of less than 120 μm .

6. A method for producing a silver-alloy sputtering target for conductive-film formation, the method comprising: subjecting a cast ingot having a component composition including In at a content in a range of 0.1 mass% to 1.5 mass% with a remainder of Ag and inevitable impurities, to a hot rolling step, a cooling step, and a machining step sequentially, thereby producing a silver-alloy sputtering target,

wherein in the hot rolling step, one or more passes of finish hot rolling are carried out under conditions where a rolling reduction rate per pass is in a range of 20% to 50%, a strain rate is in a range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of 400°C to 650°C, and

in the cooling step, quenching is carried out at a cooling rate in a range of 200 °C/min to 1000 °C/min.

7. A method for producing a silver-alloy sputtering target for conductive-film formation,
the method comprising: subjecting a cast ingot having a component composition
including In and Sn at a total content in a range of 0.1 mass% to 1.5 mass% with a
remainder of Ag and inevitable impurities, to a hot rolling step, a cooling step, and a
5 machining step sequentially, thereby producing a silver-alloy sputtering target,

wherein in the hot rolling step, one or more passes of finish hot rolling are carried
out under conditions where a rolling reduction rate per pass is in a range of 20% to 50%, a
strain rate is in a range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of
400°C to 650°C, and

10 in the cooling step, quenching is carried out at a cooling rate in a range of 200
°C/min to 1000 °C/min.

8. A method for producing a silver-alloy sputtering target for conductive-film formation,
the method comprising: subjecting a cast ingot having a component composition
15 including: In at a content in a range of 0.1 mass% to 1.5 mass%; and either one or both of
Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag
and inevitable impurities, to a hot rolling step, a cooling step, and a machining step
sequentially, thereby producing a silver-alloy sputtering target,

wherein in the hot rolling step, one or more passes of finish hot rolling are carried
20 out under conditions where a rolling reduction rate per pass is in a range of 20% to 50%, a
strain rate is in a range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of
400°C to 650°C, and

in the cooling step, quenching is carried out at a cooling rate in a range of 200
°C/min to 1000 °C/min.

9. A method for producing a silver-alloy sputtering target for conductive-film formation,

the method comprising: subjecting a cast ingot having a component composition including: In and Sn at a total content in a range of 0.1 mass% to 1.5 mass%; and either

5 one or both of Sb and Ga at a total content in a range of 0.1 mass% to 2.5 mass%, with a remainder of Ag and inevitable impurities, to a hot rolling step, a cooling step, and a machining step sequentially, thereby producing a silver-alloy sputtering target,

wherein in the hot rolling step, one or more passes of finish hot rolling are carried out under conditions where a rolling reduction rate per pass is in a range of 20% to 50%, a

10 strain rate is in a range of 3 /sec to 15 /sec, and a temperature after the pass is in a range of 400°C to 650°C, and

in the cooling step, quenching is carried out at a cooling rate in a range of 200 °C/min to 1000 °C/min.