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TITLE

ITO SPUTTERING TARGET

FIELD OF THE INVENTION

5 The present invention relates to an ITO sputtering target.

BACKGROUND OF THE INVENTION

10 A sputtering process has been heretofore known as one of thin film-forming processes. The sputtering process is a process for forming a thin film on a substrate, wherein an inert gas in a state of plasma generally made under reduced pressure is allowed to collide with a target and molecules or atoms rushing out
15 from the target by the collision energy are allowed to adhere onto a substrate to form a thin film. This process has been industrially used since film formation of large area can be made easily and films of high performance can be obtained.

20 As other sputtering processes, a reactive sputtering process wherein sputtering is carried out in a reactive gas and a magnetron sputtering process wherein a magnet is placed on a back surface of the target to achieve high-speed thin film formation are also known recently.

Of the thin films formed by the above processes, films of an oxide containing at least one of indium oxide (In_2O_3) and tin oxide (SnO_2) (referred to as "ITO" hereinafter) particularly exhibit high visible light transmission and high conductivity, and therefore they have been widely used as, for example, transparent conductive films of liquid crystal display devices, exothermic films for preventing dew condensation of glass or infrared reflecting films.

10 In the formation of a thin film of high performance through the sputtering, however, there reside the following problems.

That is to say, in the sputtering, especially in the initial stage of the sputtering, abnormal electric discharge called arcing takes place to impair film-forming stability. Further, particles adhere or accumulate on a sputtering target (sometimes referred to as a "target" simply hereinafter) to form a black deposit called nodule, and this nodule causes arcing, or new particles are produced by the arcing. Furthermore, if the particles adhere to the thin film, performance of the thin film is deteriorated, and such deterioration causes a defective thin film.

Especially in the period of time from the initiation of sputtering using a newly set sputtering target to the stoppage of occurrence of arcing (sometimes referred to as "initial arc" hereinafter) that enables manufacture of products, idling must be carried out, and this is an obstacle to improvement of the productivity.

It has been heretofore said that occurrence of such arcing or nodule is more decreased as the surface of the target is smoothed by grinding. Therefore, a surface-polished target having a smoothed surface is now the mainstream. For example, an ITO sputtering target whose surface roughness is set within the prescribed range to prevent occurrence of arcing or nodule is described in Japanese Patents No. 2750483 and No. 3152108. In order to attain such a prescribed surface roughness, however, it is necessary that after preparation of a target, rough grinding is carried out through machine grinding to control the thickness and then finish grinding (polishing) is stepwise carried out to smooth the target surface, and this results in problems of a long manufacturing time and large cost. Further, even in the use of the ITO target having a prescribed surface roughness, the initial arc cannot be prevented, and

idling must be carried out over a relatively long period of time after a new sputtering target is set.

In Japanese Patent No. 3040432, there is described a sputtering target comprising a metal compound portion and
5 a metal portion having a thin film of the metal compound formed on the metal portion, which is obtained by pre-sputtering a target constituted of a metal compound portion and a metal portion in a reactive gas-containing atmosphere. In this target, by the use of a metal
10 portion in combination, not only the cost of the target itself is decreased but also the gas released is reduced to improve thin film properties. However, there is a problem that because of the constitution of the target, the production process becomes complicated.

15 In Japanese Patent Laid-Open Publication No. 92923/1999, there is described a sputtering target having a thin film provided on the target surface. In this target, by the formation of the thin film, separation of a deposit produced in the sputtering is inhibited to
20 prevent occurrence of dust and thereby improve yield of thin film products. However, the purpose of such a constitution is to inhibit separation of a deposit produced on the target surface, and the initial particles cannot be inhibited.

Under such circumstances as described above, the present inventors have earnestly studied, and as a result, they have found that the initial particles, which are attributable to the grinding step or the like and present
5 on the target surface, or the portions, which separate from the surface layer by thermal shock of the sputtering and become particles, are main causes of arcing or nodule and that the occurrence of initial arc can be prevented by forming an ITO film having a specific film thickness
10 on a sputter surface of an ITO target. Based on the finding, the present invention has been accomplished.

OBJECT OF THE INVENTION

It is an object of the present invention to provide
15 an ITO sputtering target capable of preventing occurrence of initial arc, improving initial stability and remarkably enhancing productivity.

SUMMARY OF THE INVENTION

20 The ITO sputtering target according to the present invention comprises a target produced through a machine grinding step and made of an oxide (ITO) containing at least one of indium oxide and tin oxide and an ITO film having a film thickness of more than 1 μm , preferably not

less than 1.5 μm , more preferably not less than 2 μm , that is formed on a sputter surface of the target.

The surface roughness Ra of the ITO sputtering target of the invention may be not less than 1 μm , may be
5 not less than 1.5 μm , and may be not less than 2 μm . The term "surface roughness Ra" used herein means an arithmetic average roughness of a roughness curve measured in accordance with JIS B 0601 (1994).

In the present invention, the ITO film formed on at
10 least the sputter surface of the ITO target is preferably a film formed by sputtering.

In the present invention, an ITO film having a specific film thickness is formed on the sputter surface of the ITO target as described above, and therefore the
15 occurrence of arcing can be effectively inhibited from the initial stage of the sputtering.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a view showing a relationship between the
20 thickness of an ITO film formed on the sputter surface of the target and the cumulative frequency of arc.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is described in detail hereinafter.

An ITO target is generally produced by a sintering process comprising mixing starting material powders in a given mixing ratio, molding the mixture by various conventional dry methods or wet methods, calcining (sintering) the molded product and then subjecting the product to machine grinding.

Examples of the dry methods include CP (cold press) method, HP (hot press) method and HIP (hot isostatic press) method. According to the CP method, mixed starting material powders are charged in a mold to prepare a molded product, and the molded product is calcined (sintered) in the atmosphere or in an oxygen atmosphere. According to the HP method, mixed starting material powders are charged in a mold of an electric furnace, and they are molded and sintered at the same time with heating under pressure. According to the HIP method, mixed starting material powders or a preform is enclosed in a rubber bag or with a metal foil that forms a covering even at a high temperature, deaerated, then placed in a container and heat sintered with isotropically pressurizing with an inert medium.

The wet method is, for example, a filtration molding method described in Japanese Patent Laid-Open Publication No. 286002/1999. In the filtration molding method, a filtration mold is used. The filtration mold is a mold
5 to obtain a molded product by removing water content from a slurry of ceramic starting materials under reduced pressure, and this mold is made of a water-insoluble material. According to this method, a slurry consisting of mixed starting material powders, ion exchange water
10 and an organic additive is poured into the filtration mold, then water content is removed from the slurry under reduced pressure to prepare a molded product, and the molded product is dried, degreased and then calcined (sintered).

15 After the starting materials are molded and calcined (sintered) in a mold as described above, the sintered product is generally subjected to machine grinding to process it into a given size and then bonded to a backing plate to prepare a target.

20 For the machine grinding, an appropriate grinding method such as surface grinding, rotary polishing or blasting is adopted according to necessity. After the grinding for the above processing, the surface is subjected to rough grinding to control the thickness and

then subjected to finish grinding (sometimes referred to as "polishing" hereinafter) to further smooth the surface using grindstones having stepwise decreased grits, or subjected to blast grinding (blast grinding and finish grinding being together sometimes referred to as "polishing" hereinafter) using glass beads, alumina beads zirconia beads or the like as blasting media.

The present invention is characterized by forming an ITO film having a specific film thickness on the sputter surface of an ITO target having been subjected to the above-mentioned machine grinding. By the formation of the ITO film on the sputter surface of the target, burr or powder produced by the grinding and portions to be easily separated by thermal shock of the sputtering can be covered and joined to obtain a good target surface, and thereby occurrence of initial arc in the sputtering can be effectively reduced.

Examples of preferred methods for forming the ITO film include sputtering, deposition, ion plating, coating and spraying. Of these, sputtering is preferably used from the viewpoint of easy control of thickness and the economical viewpoint.

In the present invention, the composition ratio of the ITO film formed on the sputter surface of the target

is preferably the same as that of ITO that is a target material, but may be different as far as an element other than the constituent elements of the target material is not contained.

5 In the present invention, the film thickness of the ITO film formed on the sputter surface of the target can be measured by, for example, microscope sectional observation using a scanning electron microscope. In this specification, however, the film thickness is
10 determined in accordance with the following method. That is to say, an ITO film is formed on a glass substrate by sputtering, then the film thickness of the ITO film is measured as a difference in the height between the glass substrate and the substrate including the ITO film,
15 thereafter a film-forming rate (nm/sec) is calculated from the film thickness and the sputtering time required for the film formation, and from the sputtering time a film thickness is determined. More specifically, on a glass substrate (Corning #7059), an ITO film is formed by
20 sputtering, and a difference in the height between the substrate and the place where the ITO film is formed on the substrate is measured by means of Dektak IIA (manufactured by Sloan Technology Co.) to determine a film thickness of the ITO film. From the film thickness

of the ITO film thus determined and the sputtering time required for the film formation, a film-forming rate can be obtained.

In the present invention, the film thickness of the ITO film formed on the sputter surface of the target is preferably more than 1 μm , more preferably not less than 1.5 μm , still more preferably not less than 2 μm .

When the film thickness of the ITO film exceeds 1 μm , the cumulative frequency of arc in the sputtering is remarkably decreased, and excellent initial arc properties are obtained. The film thickness of the ITO film may be not more than 5 μm as far as it is within the above range.

In the present invention, the initial arc properties of the target can be evaluated by the use of a μ Arc Monitor (MAM Genesis, manufactured by Landmark Technology Co.) as a measuring device. That is to say, in the sputtering using a target having an ITO film formed on its sputter surface, a cumulative frequency of arc to the integrated value of electric power that is applied to a unit area of the target per unit hour (integrated applied electric power, Wh/cm^2) is measured, and based on the cumulative frequency of arc, the initial arc properties can be evaluated. It can be said that the initial arc

properties are more excellent as the cumulative frequency of arc to the integrated applied electric power decreases. Since the arc frequency of the sputtering target of the invention is low, the target has a long life and
5 excellent long-term film-forming stability.

In the present invention, by forming an ITO film having a specific film thickness on the sputter surface as described above, the initial arc can be effectively reduced, and a target capable of performing efficient
10 sputtering can be obtained even if the surface is not always subjected to mirror-smooth polishing (finish grinding) differently from the conventional process. That is to say, the surface roughness (arithmetic average roughness) Ra of the sputtering target of the invention,
15 though depends upon the surface condition of the target after machine grinding, may be larger than 0.5 μm that is conventionally considered as preferable. More specifically, the surface roughness Ra may be not less than 1 μm , may be not less than 1.5 μm , and may be not
20 less than 2 μm . As a matter of course, the present invention can also be applied to the case where the Ra of the target is not more than 0.5 μm .

Therefore, when the present invention is applied to such a case, the polishing step may be omitted, and by

forming an ITO film on a sputter surface of a target, a sputtering target can be obtained efficiently.

After the formation of the ITO film on the ITO target having been subjected to machine grinding, the target may be bonded to a backing plate in accordance with a conventional method to obtain a final sputtering target, but it is preferable to form an ITO film after the ITO target having been subjected to machine grinding is bonded to a backing plate in accordance with a conventional method. According to the latter process, the sputter surface having the ITO film formed thereon can be prevented from adhesion of impurities such as dust.

EFFECT OF THE INVENTION

According to the present invention, a sputtering target capable of effectively reducing occurrence of initial arc and having high initial stability can be obtained. By conducting sputtering using this sputtering target, formation of thin films having high performance can be efficiently carried out with enhanced productivity. Moreover, since finish grinding is omissible, the grinding step can be simplified and the cost can be decreased.

EXAMPLE

The present invention is further described with reference to the following examples, but it should be construed that the invention is in no way limited to
5 those examples.

Example 1

A In_2O_3 powder and a SnO_2 powder were mixed in a weight ratio of 90:10 ($\text{In}_2\text{O}_3:\text{SnO}_2$). From the mixture, a sintered product of ITO was prepared in accordance with a
10 conventional method, and the sintered product was used as a target material. After the target material was cut into a size of 4 inch diameter, a surface (sputter surface) to be subjected to sputtering and a surface (bonding surface) to be subjected to bonding of the
15 target material were both ground by a surface grinding machine to adjust the thickness to 6 mm. Then, the sputter surface was ground by a diamond wheel.

The target material was placed on the anode side of a sputtering device, and a different ITO target was
20 placed on the cathode side of the sputtering device. Then, sputtering was carried out under the following conditions to form an ITO film having a film thickness of 13500 Å (1.35 μm) on the sputter surface of the target placed on the anode side.

The sputtering conditions are as follows.

sputtering system: DC magnetron sputter, process
gas: Ar, process pressure: 3 mTorr, oxygen partial
pressure: 0.02 mTorr, applied electric power: 3 W/cm²

5 The surface roughness of the sputter surface of the
target was measured before and after the formation of the
ITO film in accordance with JIS B 0601 (1994). The
results are as shown in Table 1. Measurement of the
surface roughness was carried out using a surface
10 roughness meter SE1700 (manufactured by Kosaka Kenkyusho)
under the conditions of a feeler radius of 2 μ m, a feed
rate of 0.5 mm/sec, a cut-off of λ_c 0.8 mm and an
evaluation length of 4 mm.

15 Example 2

A target was prepared in the same manner as in
Example 1, except that the film thickness of the ITO film
formed on the sputter surface of the target material was
changed to 27000 Å (2.7 μ m). The surface roughness
20 before and after the formation of the ITO film was
measured in the same manner as in Example 1. The results
are set forth in Table 1.

Example 3

A target was prepared in the same manner as in Example 1, except that after the sputter surface and the bonding surface of the target material were both roughly
5 ground by a surface grinding machine to adjust the thickness to 6 mm, the sputter surface was subjected to blasting with Alundum beads instead of grinding by a diamond wheel, and the film thickness of the ITO film formed on the sputter surface of the target material was
10 changed to 27000 Å (2.7 μm). The surface roughness before and after the formation of the ITO film was measured in the same manner as in Example 1. The results are set forth in Table 1.

15 Comparative Example 1

A target was prepared in the same manner as in Example 1, except that the film thickness of the ITO film formed on the sputter surface of the target material was changed to 1350 Å (0.135 μm). The surface roughness
20 before and after the formation of the ITO film was measured in the same manner as in Example 1. The results are set forth in Table 1.

Comparative Example 2

A target was prepared in the same manner as in Example 1, except that the film thickness of the ITO film formed on the sputter surface of the target material was
5 changed to 9450 Å (0.945 μm). The surface roughness before and after the formation of the ITO film was measured in the same manner as in Example 1. The results are set forth in Table 1.

10 Test Example

Using the targets prepared in Examples 1 to 3 and Comparative Examples 1 and 2, film-forming tests were carried out under the following conditions to evaluate initial arc properties in the sputtering.

15 Sputtering conditions

sputtering system: DC magnetron sputter, process gas: Ar, process pressure: 3 mTorr, oxygen partial pressure: 0.02 mTorr, applied electric power: 3 W/cm²

The initial arc properties were evaluated based on
20 the cumulative frequency of arc to the integrated applied electric power (Wh/cm²). It can be said that the initial arc properties are more excellent as the cumulative frequency of arc to the integrated applied electric power decreases.

More specifically, the cumulative frequency of arc until the integrated electric power applied for the sputtering became 3 Wh/cm^2 was measured using a μ Arc Monitor (MAM genesis, manufactured by Landmark Technology Co.) as an arc counter under the following conditions.

Measuring conditions

detection mode: energy, arc detection voltage: 100 V, large-medium energy border: 50 mJ, hard arc minimum time: 100 μs

The results are set forth in Table 1.

Table 1

Sample	Film thickness ($\text{\AA}$)	Surface roughness (μm) before film formation	Surface roughness (μm) after film formation	Cumulative frequency of arc (number of times)
Example 1	13500	1.32	1.53	17
Example 2	27000	1.33	1.45	1
Example 3	27000	2.41	2.43	5
Comparative Example 1	1350	1.28	1.27	66
Comparative Example 2	9450	1.37	1.37	50

As can be seen from Table 1, there is not so large difference in the surface roughness Ra between before and after the formation of the ITO film on the target surface, and the Ra after the formation of the ITO film on the

target surface depends upon the surface condition of the target before the formation of the ITO film.

The evaluation results of the initial arc properties obtained when the targets prepared in Examples 1 to 3 and Comparative Examples 1 and 2 were used are shown in Fig. 1, in which the film thickness ($\text{\AA}$) is plotted as abscissa and the cumulative frequency of arc (number of times) is plotted as ordinate.

As can be seen from Table 1 and Fig. 1, the cumulative frequency of arc is effectively decreased bordering on the film thickness 10000 $\text{\AA}$ (1 μm) of the ITO film formed on the target surface, and when the film thickness of the ITO film formed on the target surface exceeds 1 μm , the initial arc properties become excellent.

What is claimed is:

1. An ITO sputtering target comprising a target produced through a machine grinding step and made of an oxide (ITO) containing at least one of indium oxide and tin oxide and an ITO film having a film thickness of more than 1 μm that is formed on a sputter surface of the target.

2. The ITO sputtering target as claimed in claim 1, wherein the film thickness of the ITO film is not less than 1.5 μm .

3. The ITO sputtering target as claimed in claim 1, wherein the film thickness of the ITO film is not less than 2 μm .

4. The ITO sputtering target as claimed in any one of claims 1 to 3, wherein the surface roughness Ra of the sputter surface is not less than 1 μm .

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5. The ITO sputtering target as claimed in any one of claims 1 to 3, wherein the surface roughness Ra of the sputter surface is not less than 1.5 μm .

6. The ITO sputtering target as claimed in any one of claims 1 to 3, wherein the surface roughness Ra of the sputter surface is not less than 2 μm .

5 7. The ITO sputtering target as claimed in any one of claims 1 to 6, wherein the ITO film formed on at least the sputter surface of the ITO target is a film formed by sputtering.