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(71) 出願人: 三井金属鉱業株式会社 (MITSUI MINING & SMELTING CO., LTD.) [JP/JP]; 〒141-8584 東京都品川区大崎一丁目11番1号 Tokyo (JP).

(72) 発明者: 仙田 貞雄 (SENDA, Sadao); 〒836-0003 福岡県大牟田市大字唐船2081 三井金属鉱業株式会社内 Fukuoka (JP). 尾野 直紀 (ONO, Naoki);

〒836-0003 福岡県大牟田市大字唐船2081 三井金属鉱業株式会社内 Fukuoka (JP). 林 博光 (HAYASHI, Hiromitsu); 〒836-0003 福岡県大牟田市大字唐船2081 三井金属鉱業株式会社内 Fukuoka (JP). 早川 泉 (HAYAKAWA, Izumi); 〒358-0032 埼玉県入間市狭山ヶ原11の10 三井研削砥石株式会社内 Saitama (JP).

(74) 代理人: 鈴木 俊一郎 (SUZUKI, Shunichiro); 〒141-0031 東京都品川区西五反田七丁目13番6号 五反田山崎ビル6階 鈴木国際特許事務所 Tokyo (JP).

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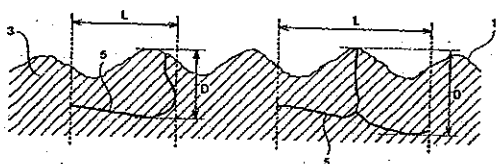
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(54) Title: SPUTTERING TARGET AND PRODUCTION METHOD THEREFOR

(54) 発明の名称: スパッタリングターゲットおよびその製造方法



(57) Abstract: A sputtering target which is produced after being gone through a mechanical grinding step, characterized in that, in its first embodiment, micro-cracks having depths and lengths of at least specified values are substantially eliminated when the sputter surface of this target is observed in section, and that, in its second embodiment, the sputter surface of a sputtering target produced after being undergone mechanical grinding is sputtered in advance before shipment. A sputtering target can be provided that effectively reduces arcing, especially, an initial arc

generation, and significantly enhances an initial stability.

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TITLE

SPUTTERING TARGET AND PROCESS FOR MANUFACTURING THE SAME

TECHNICAL FIELD

5 The present invention relates to targets used for sputtering and thin film formation and a process for manufacturing the targets.

BACKGROUND ART

10 A sputtering process has heretofore been 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
15 collide with a sputtering target (also referred to as a "target" simply hereinafter) and molecules or atoms rushing out from the target by the collision energy are allowed to deposit on a substrate to form a thin film. This process has been industrially used since film
20 formation of large area can be made easily and films of high performance can be obtained.

As other sputtering processes, there are recently known 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.

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

The target for forming the ITO film is manufactured similarly to other oxide targets. That is to say, a target material is prepared by powder metallurgy process (sintering process) which comprises molding a starting material powder and then sintering the molded product, and the target material is then subjected to surface grinding using a grinding wheel of about #200 or to other machining operations such as outline machining, to manufacture the target.

In the formation of a thin film of high performance using the sputtering target thus manufactured, 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 (sometimes referred to as "initial arc" hereinafter) takes place to impair film-forming stability and to produce particles. Further, the particles adhere to the sputtering target and accumulate thereon to form a black deposit called nodule, and this nodule causes arcing, which further induces occurrence of new particles. Furthermore, if the particles adhere to the thin film, performance of the thin film is deteriorated.

In the use of a new sputtering target, therefore, in order to avoid the above problems, idling must be carried out in the period of time from the beginning of sputtering to the stoppage of occurrence of arcing that enables manufacturing of products, even if the new sputtering target is set in the sputtering apparatus, and this is an obstacle to improvement of the productivity.

It has heretofore been said that occurrence of such arcing or nodule is more reduced as the surface of the target is smoothed by abrasion. Therefore, a surface-abraded target with a smooth surface is now the mainstream. For example, an ITO sputtering target whose surface roughness is controlled 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

5 target material, the target material is subjected rough grinding through machine grinding to adjust the thickness and then stepwise subjected to finish grinding (abrasion) to smooth the target surface, and this results in problems of a long manufacturing time and high cost.

10 Further, even in the use of the ITO target having a prescribed surface roughness, the initial arc cannot be effectively prevented, and idling must be carried out over a relatively long period of time after a new sputtering target is set in the sputtering apparatus.

15 Such problems reside also in ceramic type targets which are prepared by powder metallurgy process (sintering process) and contain, as main components, oxides other than ITO, nitrides, carbides and borides, etc., or metal type targets.

20 Under such circumstances, the present inventors have earnestly studied, and as a result, they have found that, in a sputtering target manufactured through a machine grinding step, initial particles, which are attributable to the grinding step and present on the target surface,

or portions, which separate from the surface layer by thermal shock of sputtering and become particles, are main causes of arcing or nodule, so that if these initial particles and portions are removed, occurrence of the
5 initial arc can be prevented.

The present inventors have further studied, and as a result, they have found that whether the portions separate from the surface layer by thermal shock of sputtering and become particles or not greatly depends
10 upon the presence of micro cracks formed from the surface of the target to the inside by the machine grinding, rather than the surface roughness of the target. The present inventors have also found that all of the micro cracks do not cause arcing or nodule but micro cracks
15 having a depth and a length of not less than the specific values are main causes of the initial arc, so that occurrence of the initial arc can be effectively prevented by substantially removing such micro cracks. Based on the finding, the present invention has been
20 accomplished.

Accordingly, it is an object of the invention to provide a sputtering target, which can prevent occurrence of initial arc, improve initial stability of the sputtering and remarkably enhance productivity in the

thin film formation, and a process for manufacturing the sputtering target.

DISCLOSURE OF THE INVENTION

5 The sputtering target according to a first embodiment of the present invention is a sputtering target manufactured through a machine grinding step, wherein a micro crack having a depth of not less than 15 μm and a length of not less than 40 μm , preferably a
10 depth of not less than 10 μm and a length of not less than 30 μm , is not substantially found when the sectional observation of a sputter surface of the target is made. In the sputtering target, the number of micro cracks having a depth of not less than 5 μm and less than 10 μm
15 and a length of not less than 10 μm and less than 30 μm is preferably 5 or less based on 2.5 mm of a length in the width direction of the section.

 The sputtering target of the first embodiment of the invention is preferably one obtained by subjecting its
20 sputter surface to any one of sputtering treatment, laser treatment and dry etching treatment.

 The sputtering target of the first embodiment of the invention preferably comprises a target material prepared by powder metallurgy process, and preferably contains at

least one of indium oxide and tin oxide as a main component.

In the sputtering target of the first embodiment of the invention, the surface roughness Ra of the sputter
5 surface may be not less than 1.0 μm .

The sputtering target according to a second embodiment of the present invention is a sputtering target manufactured through a machine grinding step, a sputter surface of which has been subjected to sputtering
10 treatment prior to shipping.

In the sputtering target of the second embodiment of the invention, the integrated electric energy applied to the sputter surface of the sputtering target in the sputtering treatment is preferably not less than 0.005
15 Wh/cm^2 , more preferably not less than 0.01 Wh/cm^2 , still more preferably not less than 0.02 Wh/cm^2 .

In the sputtering target of the second embodiment of the invention, a surface protective film has been preferably stuck to the sputter surface of the sputtering
20 target when the target is shipped.

The sputtering target of the second embodiment of the invention is preferably one manufactured by a sintering process.

The sputtering target of the second embodiment of the invention preferably contains indium oxide as a main component, or is preferably an oxide (ITO) containing at least one of indium oxide and tin oxide.

5 The process for manufacturing the sputtering target of the second embodiment of invention comprises machine grinding a surface of a target, preferably a surface of a target obtained by a sintering process, and then, prior to shipping, subjecting at least a sputter surface of the
10 target to sputtering treatment.

When the sputtering target of the second embodiment of the invention is an ITO sputtering target, the process for manufacturing the ITO sputtering target comprises machine grinding a surface of an ITO target obtained by
15 sintering a material containing indium oxide and tin oxide as main components and then, prior to shipping, subjecting at least a sputter surface of the ITO target to sputtering treatment.

20

BRIEF DESCRIPTION OF THE DRAWING

Fig. 1 is a schematic view to explain examples of micro cracks which can be found when the sectional observation of the sputter surface of the target is made.

BEST MODE FOR CARRYING OUT THE INVENTION

The present invention is described in detail hereinafter.

The first embodiment and the second embodiment of
5 the sputtering target according to the invention are both manufactured through a machine grinding step.

That is to say, the sputtering target according to the invention has only to be a sputtering target obtained through a machine grinding step. The target material for
10 use in the invention is not particularly restricted, and for example, it may be a ceramic type target containing as a main component an oxide, a nitride, a carbide, a boride or the like, or may be a target made of a metal. Examples of such target materials include those (ITO)
15 containing at least one of In_2O_3 and SnO_2 as a main component, those (IZO) containing at least one of In_2O_3 and ZnO as a main component, $\text{ZnO-Al}_2\text{O}_3$, In_2O_3 , SnO_2 , ZnO , Al_2O_3 , SiO_2 , Ta_2O_5 , MgO , NiO , Si_3N_4 , AlN , SiC , Mo , W , Cr , Ti , Zr , Hf , Nb , Ta and Al . Of these, the ITO sputtering
20 targets are particularly effectively applicable to the first and the second embodiments of the invention because high initial stability is required in order to carry out efficient sputtering.

The process to prepare the target material is not particularly restricted, and for example, the ceramic type target can be prepared by a sintering process (powder metallurgy process) wherein a mixture of starting material powders obtained by mixing starting materials in a given ratio or a homogeneous mixture of starting material powders obtained by co-precipitation method or the like is molded by various dry methods or wet methods conventionally known, then subjected to sintering and then subjected to machine grinding. In case of the metal type target, a vacuum dissolution process wherein the mixture of starting material powders is vacuum dissolved and then subjected to casting, plastic working and machine grinding, or powder metallurgy process wherein the mixture of starting material powders is molded by HIP (hot isostatic press) method or CIP (cold isostatic press) method and then subjected to sintering and plastic working is employable.

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, a mixture of starting material powders is 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, a mixture of starting material powders is charged in a mold of an electric furnace and then subjected to molding and sintering at the same time with heating under pressure. According to
5 the HIP method, a mixture of 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 sintered by heating with isotropically pressurizing with an inert
10 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
15 made of a water-insoluble material and to obtain a molded product by removing water content from a slurry of ceramic starting materials under reduced pressure. According to this method, a slurry consisting of a mixture of starting material powders, ion exchange water
20 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 sintered.

The calcining temperature in each of the above methods is desirably a temperature appropriately determined according to the starting materials used.

After the starting materials are molded in a mold
5 and sintered to prepare a target material as described above, the target material is subjected to machine grinding, such as surface grinding, in order to form it into a given size or to smooth the surface. That is to say, the sputtering target is generally manufactured
10 through at least a machine grinding step.

It is thought that on the surface of the target manufactured through the machine grinding step as described above, initial particles attributable to the grinding step or portions which separate from the surface
15 layer by thermal shock of sputtering and become particles are usually present, and these particles adhere to the substrate during the sputtering to cause thin film defect or adhere to the target again and accumulate thereon to form a black deposit called nodule, which causes arcing
20 to thereby further form new particles.

In the present invention, therefore, by subjecting the sputter surface of the sputtering target manufactured through the machine grinding step to the later-described specific treatment, the particles and the portions which

are liable to separate by thermal shock of sputtering are removed, whereby the initial arc is effectively reduced.

First embodiment of the invention

First, the first embodiment of the present invention
5 is described. The sputtering target according to the first embodiment of the invention is characterized in that the sputtering target is manufactured through a machine grinding step in accordance with the conventional process as described above, and thereafter or in the
10 course of the manufacturing, the sputtering target is subjected to the later-described treatment to substantially remove micro cracks.

Since the machine grinding is a technique to grind a workpiece by cutting it with an abrasive grain cutting
15 edge of a grinding wheel that is rotating at a high speed, and because of the contact stress with the abrasive grain, cracks are formed on a surface (particularly surface to be subjected to sputtering, referred to as "sputter surface" hereinafter) of the target and inside the target.
20 Of the cracks, those observed from the surface have been recognized as grinding damage or machining defect, but fine cracks inwardly extending from the sputter surface (referred to as "micro cracks" hereinafter) have not been studied yet.

The micro cracks occur inevitably so far as the machine grinding is carried out, though the degree of occurring the micro cracks such as the shapes (depth, length, etc.) and the number etc. vary. The degree of
5 occurring the micro cracks is influenced by weight and speed of the grinding wheel used in the machine grinding, shape of the abrasive grain, material of the workpiece, etc.

In the first embodiment of the invention, such micro
10 cracks are paid attention, and by substantially removing micro cracks having a depth and a length of not less than the specific values, a target effectively preventing occurrence of initial arc can be provided.

That is to say, all of the micro cracks do not cause
15 the initial arc but micro cracks having a depth and a length of not less than the specific values cause the initial arc, as previously described, so that by substantially removing such specific micro cracks so that they cannot be found when the sectional observation of
20 the sputter surface of the target is made, occurrence of the initial arc can be effectively prevented.

Specifically, the sputtering target according to the first embodiment of the invention is a sputtering target manufactured through a machine grinding step, wherein a

micro crack having a depth of not less than 15 μm and a length of not less than 40 μm , preferably a depth of not less than 10 μm and a length of not less than 30 μm , is not substantially found when the sectional observation of a sputter surface of the target is made.

The expression "a micro crack is not substantially found" used herein means that removal of micro cracks is carried out on the target until no micro crack having a depth and a length of not less than the specific values can be found when the sectional observation of the sputter surface of the target is made.

Specifically, it is meant that a micro crack having a depth of not less than 15 μm and a length of not less than 40 μm , preferably a depth of not less than 10 μm and a length of not less than 30 μm , is not substantially found in the region of a length of 2.5 μm in the width direction when the sectional observation of the sputter surface of the target is made. This means that even if a micro crack having a depth and a length of less than the above values is present, this micro crack itself does not cause arcing and does not become a source of particles either because it does not bring about chipping by thermal shock of sputtering.

More specifically, it is desirable that when the sectional observation of the sputter surface of the target is made, a micro crack having a depth and a length of not less than the specific values is not found, and
5 the number of micro cracks having a depth of not less than 5 μm and less than 10 μm and a length of not less than 10 μm and less than 30 μm is 5 or less, preferably 3 or less, more preferably 1 or less, based on 2.5 mm of a length in the width direction of the section.

10 As a matter of course, it is most desirable that when the sectional observation of the sputter surface of the target is made, even a micro crack having a depth of not less than 5 μm and less than 10 μm and a length of not less than 10 μm and less than 30 μm is not found,
15 that is, the number of the above-mentioned micro cracks is 0 based on 2.5 mm of a length in the width direction of the section.

However, when the number of the micro cracks is 5 or less, preferably 3 or less, more preferably 1 or less,
20 the initial arc can be effectively reduced, and there is no trouble in the sputtering.

The means for the sectional observation of the sputter surface of the target is, for example, microscopic observation using an optical microscope, an

electron microscope or a scanning electron microscope (SEM).

The sputter surface of the target has fine protrusions and depressions because the target has passed through the machine grinding step as previously described, and how to define the depth and the length of a micro crack comes into question. Therefore, definitions of the depth and the length of a micro crack in the present specification are explained below with reference to Fig.

10 1.

In the present specification, the depth of a micro crack is, as shown in Fig. 1, a depth D from the top of the highest mountain in the ridgeline formed by the sputter surface 1 corresponding to the region where the micro crack 5 is present and the section 3 to the deepest position of the micro crack 5.

In the present specification, the length of a micro crack is, as shown in Fig. 1, a maximum length L of the micro crack 5 in the horizontal direction on the section 3.

The sputtering target of the first embodiment of the invention has only to be a target substantially free from a micro crack having a depth and a length of not less than the specific values, and the surface roughness

(arithmetic mean roughness of roughness curve) R_a
(measured in accordance with JIS B 0601 (1994)) of the
sputter surface of the target may be not less than $1.0\ \mu\text{m}$,
or may be not less than $1.5\ \mu\text{m}$, or may be not less than
5 $2.0\ \mu\text{m}$.

In the sputtering target of the first embodiment of
the invention, accordingly, the surface of the target
does not always need to be abraded smoothly in order to
lower the surface roughness, differently from the
10 conventional targets, so that such an abrasion step can
be omitted. Hence, the production process can be
simplified, and the productivity of the target can be
enhanced.

However, it is a matter of course that R_a of less
15 than $1.0\ \mu\text{m}$ can be applied to the present invention.

In order to obtain the sputtering target of the
first embodiment of the invention, namely, a target
substantially free from a micro crack having a depth and
a length of not less than the specific values, it is
20 necessary to carry out removal of micro cracks until such
a specific micro crack cannot be found as described above.

As the method to substantially free the target from
the micro cracks (removal of micro cracks), any method is
adoptable provided that the method can substantially

remove the micro cracks. For example, precision grinding in which the weight or the rotational speed of a grinding wheel is strictly controlled, sputtering treatment, laser treatment, dry etching treatment and blasting treatment
5 can be carried out on the sputter surface of the target.

Of these, sputtering treatment, laser treatment and dry etching treatment are preferable from the viewpoint of excellent productivity of the target.

The removal of micro cracks is described below
10 giving as an example a method of subjecting the sputter surface of the target to sputtering treatment.

When the sputtering treatment is adopted as a method to substantially free the target from the micro cracks (removal of micro cracks), the sputtering conditions,
15 such as mode of sputtering, sputtering gas and gas pressure, are not particularly restricted, and they can be appropriately selected according to necessity. For example, it is preferable to use, as a sputtering gas, an inert gas such as argon and if necessary an oxygen gas
20 and to set the gas pressure to 1 to 10 mTorr.

The integrated electric energy (Wh/cm^2) applied in the sputtering treatment, that is, integrated electric energy per unit area of the target, which is applied in

the sputtering treatment, is in the range of preferably 0.1 to 10 Wh/cm², more preferably 0.5 to 5 Wh/cm².

When the integrated electric energy applied is in the above range, the micro cracks can be substantially removed, and the time required for manufacturing the target and the productivity such as production cost are excellent.

Specifically, a target (which may be bonded to a backing plate) manufactured through a machine grinding step is set in a sputtering apparatus using DC magnetron sputtering system, and sputtering is carried out using a sputtering gas of the above composition until the aforesaid integrated electric energy is reached, whereby the sputtering target of the first embodiment of the invention can be obtained.

In the target thus obtained, a micro crack having a depth and a length of not less than the specific values is not substantially found when the sectional observation of the sputter surface is made, and when this target is used for thin film formation utilizing sputtering, the target can effectively prevent occurrence of initial arc and exhibits excellent initial arc properties.

The initial arc properties of the sputtering target of the first embodiment of the invention can be evaluated

based on the cumulative frequency of arc occurrence to the integrated electric energy (Wh/cm^2) applied when thin film formation is carried out using the target having been subjected to the above treatment, that is, when
5 sputtering is carried out using such a target. The cumulative frequency of arc occurrence is measured by the use of a μ Arc Monitor (MAM Genesis, manufactured by Landmark Technology Co.). In the use of the sputtering target according to the first embodiment of the invention,
10 the cumulative frequency of arc occurrence is low, and the sputtering target exhibits excellent initial arc properties.

Second embodiment of the invention

Next, the second embodiment of the present invention
15 is described.

The sputtering target according to the second embodiment of the invention is characterized in that the sputter surface of the target obtained after machine grinding of a target material has been subjected to
20 sputtering treatment prior to shipping from the sputtering target maker.

As previously described, it is thought that the cause of the initial arc is that the particles or the portions which are liable to separate by the thermal

shock of sputtering are present on the sputter surface of the sputtering target manufactured through a machine grinding step.

Therefore, if the particles or the portions which
5 are liable to separate by the thermal shock of sputtering can be removed by previously subjecting the sputter surface itself of the sputtering target to sputtering treatment, the initial arc can be effectively reduced.

Also in this case, it is thought that whether the
10 portions separate by the thermal shock of sputtering or not depends upon the presence of micro cracks formed from the surface of the target to the inside by the machine grinding, particularly the presence of micro cracks having a depth and a length of not less than the specific
15 values, similarly to the first embodiment of the invention.

Also in the second embodiment of the invention, therefore, it is desirable that the sputter surface of the sputtering target is previously subjected to
20 sputtering treatment to give a target substantially free from a micro crack having a depth and a length of not less than the specific values, similarly to the first embodiment of the invention. However, if performance to

such an extent is not required, the sputtering target is not limited thereto.

That is to say, in the second embodiment of the invention, the integrated electric energy applied to the sputter surface in the sputtering treatment previously conducted is preferably not less than 0.005 Wh/cm^2 , more preferably not less than 0.01 Wh/cm^2 , still more preferably not less than 0.02 Wh/cm^2 , particularly preferably not less than 0.1 Wh/cm^2 . The term "integrated electric energy (Wh/cm^2)" used herein means an integrated electric energy per unit area, which is applied to the sputter surface of the target in the sputtering treatment conducted prior to shipping.

When the integrated electric energy is not less than 0.005 Wh/cm^2 , the target having been previously subjected to the sputtering treatment exhibits excellent initial arc properties and can effectively prevent occurrence of the initial arc. On the user side of the sputtering target, therefore, sputtering for thin film formation can be immediately carried out by the use of the sputtering target without substantially conducting idling, so that the productivity in the thin film formation can be enhanced and the thin film formation can be efficiently carried out.

The sputtering conditions for the sputtering treatment previously carried out are not particularly restricted, and sputtering is carried out under the usual conditions until the above-mentioned integrated electric energy is reached.

In the second embodiment of the invention, the upper limit of the integrated electric energy is not particularly restricted, but from the viewpoints of the time required for manufacturing the sputtering target and the productivity such as cost, the integrated electric energy is desirably not more than 10 Wh/cm^2 .

The initial arc properties of the target can be evaluated based on the integrated electric energy (Wh/cm^2) applied until the interval of arcing occurrence becomes not less than 10 seconds in the case where sputtering is carried out using a target having a diameter of 6 inches. Specifically, sputtering is carried out to detect arc by the use of an arc counter (manufactured by Landmark Technology Co.), and the integrated electric energy applied until the initial arc of the target having a diameter of 6 inches converges, that is, the integrated electric energy applied until the interval between occurrence of arcing and occurrence of next arcing becomes not less than 10 seconds is measured.

Based on the measured value of the integrated electric energy, the initial arc properties are evaluated. As the value of the integrated electric energy is decreased or as the cumulative frequency of arc occurrence is lowered, the initial arc properties of the target become more excellent.

In the second embodiment of the invention, the sputter surface of the target is previously subjected to sputtering, so that the surface roughness Ra of the sputter surface of the target is usually larger than the initial value of the target measured immediately after the machine grinding. Therefore, Ra of the sputtering target of the second embodiment of the invention may be more than 0.5 μm that has been conventionally considered as preferable. As a matter of course, Ra of not more than 0.5 μm is applicable to the second embodiment of the invention.

In the present specification, the surface roughness Ra means an arithmetic mean roughness of a roughness curve measured in accordance with JIS B 0601 (1994).

More specifically, when sputtering is continued, the Ra value of the sputtering target of the second embodiment of the invention converges to an almost constant value regardless of the initial value, and from

the viewpoint of productivity in the sputtering treatment, the Ra value of the sputtering target may be in the range of 0.1 to 5.0 μm , or may be in the range of 0.1 to 3.0 μm .

The sputtering target of the second embodiment of
5 the invention is preferably one having a surface protective film which is stuck to the sputter surface immediately after the sputter surface is subjected to sputtering treatment. By sticking the surface protective film to the sputter surface, adhesion of impurities or
10 adsorption of gas on the target surface can be prevented.

The surface protective film has only to be stuck to at least the sputter surface so that adhesion of impurities or adsorption of gas on the target surface can be prevented, but the surface protective film may be
15 naturally stuck to the whole of the target. The surface protective film can be stuck by bonding a resin film to the sputter surface or vacuum packaging the whole of the target with a resin film. Of these, the vacuum packaging is preferable because bubbles hardly remain between the
20 film and the target. The resin film used for the surface protective film is not particularly restricted, but in order to prevent transfer of particles onto the sputter surface, a resin film containing no releasable particles is preferable.

Next, the process for manufacturing the sputtering target according to the second embodiment of the invention is described.

The process for manufacturing the sputtering target according to the second embodiment of the invention is characterized in that a surface of a target, preferably a surface of a target obtained by a sintering process, is subjected to machine grinding, and then, prior to shipping, at least the sputter surface of the target is subjected to sputtering treatment.

When the sputtering target according to the second embodiment of the invention is an ITO sputtering target, the process for manufacturing the ITO sputtering target is characterized in that a surface of the ITO target obtained by sintering a starting material containing indium oxide and tin oxide as main components is subjected to machine grinding, and then, prior to shipping, at least the sputter surface of the ITO target is subjected to sputtering treatment.

As the process for manufacturing the sputtering target of the second embodiment of the invention, a process for manufacturing a general sputtering target is employable except that the sputter surface of the sputtering target is subjected to sputtering treatment.

That is to say, the starting material is molded in a mold and sintered by a sintering process (powder metallurgy process) or cast by a vacuum dissolution process to prepare a target material, and the target
5 material is then subjected to machine grinding, such as surface grinding, in order to form it into a given size or to smooth the surface. After the machine grinding, the target is usually bonded to a backing plate to prepare a sputtering target.

10 For the machine grinding, an appropriate grinding method, such as surface grinding, rotary abrasion or blasting, is adopted according to necessity. After the grinding for the above forming, the surface is subjected to rough grinding to adjust the thickness and then
15 subjected to finish grinding (sometimes referred to as "abrasion" hereinafter) using grinding wheels having stepwise decreased grits to further smooth the surface, or subjected to blasting (blasting and finish grinding being together sometimes referred to as "abrasion"
20 hereinafter) using glass beads, alumina beads zirconia beads or the like as blasting media.

In the process for manufacturing the sputtering target of the second embodiment of the invention, the surface of the target is machine ground as described

above, and then, prior to shipping, at least the sputter surface of the target is subjected to sputtering treatment to manufacture the sputtering target. This sputtering treatment may be carried out after rough grinding, or may be carried out after finish grinding or blasting or after bonding to backing plate.

It is thought that by subjecting at least the sputter surface to sputtering as described above, flash and chipping powder formed by the grinding or portions liable to separate by thermal shock of sputtering can be removed, and as a result, the initial arc can be effectively reduced.

According to the second embodiment of the invention, therefore, a target capable of reducing initial arc and conducting efficient sputtering can be obtained even if the surface of the target is not always subjected to mirror-smooth abrasion or even if blasting with beads of fine grits to lower the surface roughness is not conducted. That is to say, in the application of the second embodiment of the invention to a target, a sputtering target can be obtained by subjecting the sputter surface of a target to sputtering treatment without conducting an abrasion step.

EFFECT OF THE INVENTION

According to the first embodiment of the present invention, occurrence of arcing derived from micro cracks formed by the machine grinding, particularly occurrence
5 of initial arc, can be effectively reduced, and the initial stability can be remarkably improved. Further, since the abrasion step is omissible, the production process can be simplified and the productivity of the target can be enhanced.

10 According to the second embodiment of the present invention, a sputtering target capable of effectively reducing occurrence of initial arc and enhancing initial stability can be obtained. By conducting sputtering using this sputtering target, thin film formation of high
15 performance can be efficiently carried out with high productivity. Further, since the abrasion step is omissible, the grinding process can be simplified and can be carried out at low cost.

20

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 those examples.

First, examples of the first embodiment of the present invention and comparative examples are described.

Preparation Example A1

Preparation of target

5 The targets used in Examples A1 to A3 and Comparative Example A1 were prepared in the following manner.

 An In_2O_3 powder and a SnO_2 powder were mixed in a ratio of 90:10 (In_2O_3 : SnO_2) by wt%. From the mixture, a
10 sintered product of ITO was prepared in accordance with a conventional method, and the sintered product was used as a target material. The target material had a relative density of 99.7%.

 The target material was cut to give 8 pieces having
15 a diameter of 101.6 mm. Then, they were set on the same surface grinder, and both of a sputter surface and a surface to be bonded (bonding surface) of each piece were ground with a diamond grinding wheel of #170 to obtain targets No. a1 to a8 each having a thickness of 6 mm.

20

Example A1

Removal of micro cracks

 The targets No. a1 and a2 obtained in Preparation Example A1 were each bonded to a backing plate made of

copper. Then, the targets were set in a sputtering apparatus (EX-3013M, manufactured by Shinkuu Kikai Kogyo K.K.) and subjected to sputtering treatment for the removal of micro cracks under the following conditions.

5 Sputtering conditions

Sputtering system: DC magnetron sputtering

Process gas: Ar

Process pressure: 3 mTorr

Oxygen partial pressure: 0.02 mTorr

10 Electric energy applied: 3 W/cm^2

Integrated electric energy applied: 10 Wh/cm^2

The surface roughness of the sputter surface of each of the targets No. a1 and a2 was measured before and after the sputtering treatment by the use of a surface
15 roughness meter SE1700 (manufactured by Kosaka Kenkyusho) under the conditions of a feeler radius of $2 \mu\text{m}$, a feed rate of 0.5 mm/sec , a cut-off of $\lambda_c 0.8 \text{ mm}$ and an evaluation length of 4 mm in accordance with JIS B 0601 (1994). The results are set forth in Table 1.

20 Sectional observation of target

Using a grinder (manufactured by Okamoto Kosaku Kikai K.K.), the target No. a1 having been subjected to the sputtering treatment was cut in the thickness

direction with a diamond #180 cutting blade having a diameter of 205 mm and a thickness of 1 mm.

Then, using a grinder (manufactured by Okamoto Kosaku Kikai K.K.), the cut surface of the target No. a1
5 was ground with a #600 diamond grinding wheel having a diameter of 205 mm and a thickness of 10 mm.

Then, using a single side lapping machine (manufactured by Speedfam Co.), the cut surface of the target No. a1 was lapped with loose abrasive grains of
10 GC#1000.

Then, using a single side lapping machine (manufactured by Speedfam Co.), the cut surface of the target No. a1 was abraded with diamond abrasive grains having an average grain diameter of 0.1 μm .

15 The cut surface of the target No. a1 was observed by means of an optical microscope (BX5-33P (equipped with a reflected light illumination device), manufactured by Olympus Optical Co., Ltd.). The result is set forth in Table 1.

20 Evaluation of initial arc properties

The target No. a2 having been subjected to the sputtering treatment for the removal of micro cracks similarly to the target No. a1 was set in a sputtering apparatus (EX-3013M, manufactured by Shinkuu Kikai Kogyo

K.K.) and subjected to sputtering for the thin film formation under the following conditions to detect arcing generated in the sputtering by the use of a μ Arc Monitor (MAM Genesis, manufactured by Landmark Technology Co.).

- 5 The sputtering conditions and the arcing detection conditions are as follows. The result is set forth in Table 1.

Sputtering conditions

Sputtering system: DC magnetron sputtering

- 10 Process gas: Ar

Process pressure: 3 mTorr

Oxygen partial pressure: 0.02 mTorr

Electric energy applied: 3 W/cm²

Integrated electric energy applied: 5 Wh/cm²

- 15 μ Arc Monitor measuring conditions

Detection mode: energy

Arc detection voltage: 100 V

High-medium energy boundary: 50 mJ

Hard arc minimum time: 100 μ s

20

Example A2

The procedure of Example A1 was repeated except that the targets No. a3 and a4 were used instead of the targets No. a1 and a2, and of the sputtering conditions,

the integrated electric energy applied in the sputtering treatment for the removal of micro cracks was changed to 6 Wh/cm² from 10 Wh/cm². The results are set forth in Table 1.

5

Example A3

The procedure of Example A1 was repeated except that the targets No. a5 and a6 were used instead of the targets No. a1 and a2, and of the sputtering conditions, 10 the integrated electric energy applied in the sputtering treatment for the removal of micro cracks was changed to 3 Wh/cm² from 10 Wh/cm². The results are set forth in Table 1.

15 Comparative Example A1

The procedure of Example A1 was repeated except that the targets No. a7 and a8 were used instead of the targets No. a1 and a2, and the removal of micro cracks was not carried out. The results are set forth in Table 20 1.

	Target No.	Number of micro cracks *			Surface roughness before sputtering (removal of cracks) Ra (μm)	Surface roughness after sputtering (removal of cracks) Ra (μm)	Frequency of arc occurrence (times)
		D $\geq$ 15	D $\geq$ 10	D $\geq$ 5			
		L $\geq$ 40	L $\geq$ 30	L $\geq$ 10			
Ex. A1	a1	0	0	0	1.1	1.3	-
	a2	-	-	-	1.2	1.2	1
Ex. A2	a3	0	0	1	1.1	1.1	-
	a4	-	-	-	1.0	1.2	2
Ex. A3	a5	0	0	3	1.1	1.1	-
	a6	-	-	-	1.0	1.1	7
Comp. Ex. A1	a7	5	8	10	1.1	-	-
	a8	-	-	-	1.2	-	21

*: D is a depth (μm) of a micro crack.

L is a length (μm) of a micro crack.

5

Next, examples of the second embodiment of the present invention are described.

Example B1

An In_2O_3 powder and a SnO_2 powder were mixed in a ratio of 90:10 (In_2O_3 : SnO_2) by wt%. From the mixture, a sintered product of ITO was prepared in accordance with a conventional method, and the sintered product was used as a target material. After the target material was cut into pieces having a diameter of 6 inches, both of a surface to be sputtered (sputter surface) and a surface

15

to be bonded (bonding surface) of each piece were ground with a surface grinder to adjust the thickness to 5 mm. Then, the sputter surfaces of the pieces were ground with diamond grinding wheels of different grits to obtain
5 targets No. b1 to b4. Then, a target No. b5 was prepared in the same manner as described above except that the sputter surface was subjected to blasting using Alundum as a blasting medium instead of grinding with a diamond grinding wheel.

- 10 Then, the targets were each bonded to a backing plate made of copper, then set in a homemade sputtering apparatus and subjected to sputtering under the following conditions.

Sputtering conditions

- 15 Sputtering system: DC magnetron sputtering
Process gas: Ar
Process pressure: 3 mTorr
Oxygen partial pressure: 0.03 mTorr
Electric energy applied: 1.64 W/cm²

- 20 In the above sputtering operation, detection of arc was carried out using a μ Arc Monitor (MAM Genesis, manufactured by Landmark Technology Co.) as an arc counter under the measuring conditions of a detection mode of energy, an arc detection voltage of 100 V, a

high-medium energy boundary of 50 mJ and a hard arc minimum time of 100 μ s, and the integrated electric energy applied until the initial arc converged (until the arc interval became not less than 10 seconds) was

5 measured. Then, the sputtering was continued until the integrated electric energy became 0.1 Wh/cm². The results are set forth in Table 2.

Further, the surface roughness Ra of the sputter surface of each target before the sputtering was measured
10 in accordance with JIS B 0601 (1994). The results are set forth in Table 2. The measurement of surface roughness was made using a surface 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
15 cut-off of λ c 0.8 mm and an evaluation length of 4 mm.

Table 2

Sample No.	Relative Density (%)	Density (g/cm ³)	Surface roughness Ra before sputtering treatment (μm)	Integrated electric energy applied (Wh/cm ²) *1	Frequency of arc occurrence (times) *2
b 1	99.1	7.09	0.35	0.0100	261
b 2	99.5	7.12	1.38	0.0120	1119
b 3	99.1	7.09	1.01	0.0073	740
b 4	99.3	7.10	0.79	0.0091	83
b 5	99.6	7.13	2.47	0.0064	1052

*1: integrated electric energy applied until the interval
5 of arc occurrence becomes not less than 10 seconds

*2: frequency of arc occurrence until the interval of arc
occurrence becomes not less than 10 seconds

10 Example B2

The samples used in Example B1 were taken out of the
homemade sputtering apparatus, then subjected to vacuum
packaging and allowed to stand for one day. Thereafter,
the samples were taken out of the vacuum packages, set in
15 the above-mentioned homemade sputtering apparatus and
subjected to sputtering under the same sputtering
conditions as in Example B1 until the integrated electric
energy became 5 Wh/cm² including the integrated electric
energy of Example B1. Further, the surface roughness Ra

before the sputtering treatment was measured in the same manner as in Example B1. The results are set forth in Table 3.

5

Table 3

Sample No.	Integrated electric energy applied before sputtering (Wh/cm ²)	Surface roughness Ra before sputtering treatment (μm)	Integrated electric energy applied (Wh/cm ²) *1	Frequency of arc occurrence (times) *2
b 1	0.1	0.37	0.0004	2
b 2	0.1	1.35	0.0009	2
b 3	0.1	1.09	0.0005	3
b 4	0.1	0.81	0.0009	4
b 5	0.1	2.48	0.0004	2

*1: integrated electric energy applied until the interval of arc occurrence becomes not less than 10 seconds

10 *2: frequency of arc occurrence until the interval of arc occurrence becomes not less than 10 seconds

It can be seen from Table 3 that the samples having been subjected to the sputtering treatment had excellent
 15 initial arc properties because convergence of arc immediately after sputtering was rapid and the frequency of arc occurrence was decreased.

Example B3

The samples used in Example B2 were taken out of the homemade sputtering apparatus, then set therein again and subjected to sputtering under the same sputtering conditions as in Example B1 until the integrated electric energy became 6 Wh/cm² including those of Example B1 and Example B2. As a result, any target was free from occurrence of arcs with an arc interval of less than 10 seconds. Further, the surface roughness Ra before the sputtering treatment was measured in the same manner as in Example B1. The results are set forth in Table 4.

Table 4

Sample No.	Integrated electric energy applied before sputtering (Wh/cm ²)	Surface roughness Ra before sputtering treatment (μm)	Integrated electric energy applied (Wh/cm ²) *1	Frequency of arc occurrence (times) *2
b 1	5.0	2.01	0	0
b 2	5.0	1.74	0	0
b 3	5.0	2.23	0	0
b 4	5.0	2.92	0	0
b 5	5.0	2.36	0	0

*1: integrated electric energy applied until the interval of arc occurrence becomes not less than 10 seconds

*2: frequency of arc occurrence until the interval of arc occurrence becomes not less than 10 seconds

INDUSTRIAL APPLICABILITY

The sputtering target according to the invention can effectively reduce occurrence of initial arc, and hence
5 it can be favorably used as a target used for sputtering and thin film formation.

What is claimed is :

1. A sputtering target, comprising a target material of ITO containing at least one or indium oxide and tin oxide as a main component prepared by powder metallurgy process, which is manufactured through a machine grinding step followed by subjecting its sputter surface to sputtering treatment or dry etching treatment, wherein the surface roughness Ra of the sputter surface of the target is not less than 1.0 μm and a micro crack having a depth of not less than 15 μm and a length of not less than 40 μm is not substantially found when the sectional observation of the sputter surface of the target is made.

2. The sputtering target as claimed in claim 1, wherein a micro crack having a depth of not less than 10 μm and a length of not less than 30 μm is not substantially found when the sectional observation of a sputter surface of the target is made.

3. The sputtering target as claimed in claim 2, wherein the number of micro cracks having a depth of not less than 5 μm and less than 10 μm and a length of not less than 10 μm and less than 30 μm is 5 or less based on 2.5 mm of a length in the width direction of the section when the sectional observation of a sputter surface of the target is made.

4. A sputtering target manufactured through a machine grinding step, a sputter surface of which has been subjected to sputtering treatment prior to shipping.

5. The sputtering target as claimed in claim 4, wherein the integrated electric energy applied to the sputter surface of the sputtering target in the sputtering treatment is not less than 0.005 Wh/cm^2 .

6. The sputtering target as claimed in claim 4, wherein the integrated electric energy applied to the sputter surface of the sputtering target in the sputtering treatment is not less than 0.01 Wh/cm^2 .

7. The sputtering target as claimed in claim 4, wherein the integrated electric energy applied to the sputter surface of the sputtering target in the sputtering treatment is not less than 0.02 Wh/cm^2 .

8. The sputtering target as claimed in any one of claims 4 to 7, wherein a surface protective film has been stuck to the sputter surface of the sputtering target when the target is shipped.

9. The sputtering target as claimed in any one of claims 4 to 8, which is manufactured by a sintering process.

10. The sputtering target as claimed in any one of claims 4 to 9, which contains indium oxide as a main component.

11. The sputtering target as claimed in any one of claims 4 to 9, which is an oxide (ITO) containing at least one of indium oxide and tin oxide.

12. A process for manufacturing a sputtering target, comprising machine grinding a surface of a target and then, prior to shipping, subjecting at least a sputter surface of the target to sputtering treatment.

13. The process for manufacturing a sputtering target as claimed in claim 12, wherein the target is obtained by a sintering process.

14. A process for manufacturing a sputtering target, comprising machine grinding a surface of an ITO target obtained by sintering a material containing indium oxide and tin oxide as main components and then, prior to shipping, subjecting at least a sputter surface of the ITO target to sputtering treatment.

ABSTRACT

The sputtering target of the present invention is a sputtering target manufactured through a machine grinding step, and a first embodiment of the invention is

5 characterized in that a micro crack having a depth and a length of not less than the specific values is not substantially found when the sectional observation of the sputter surface of the target is made. A second

10 embodiment of the invention is characterized in that the sputter surface of the sputtering target manufactured through a machine grinding step has been subjected to sputtering treatment prior to shipping. According to the invention, a sputtering target capable of effectively

15 reducing occurrence of arcing, particularly occurrence of initial arc, and capable of remarkably improving initial stability can be provided.