

**TARGET OF SPUTTERING FOR FORMING
PROTECTIVE FILM ON OPTICAL RECORDING
MEDIA AND PROCESS FOR PREPARING THE TARGET** ✓

Background of the Invention

5 The present invention relates to a target of sputtering for forming a protective film on an optical recording medium (hereinafter referred to as an optical recording protective film) and a process for preparing the same. More particularly, it relates to a sputtering target for optical recording protective film formation, which is prepared by high-temperature hot pressing using fine SiO_2 powder, which has high strength and generates little dust or
10 particulate matter during sputtering and therefore stands up to long-term use, and a process for preparing such a target.

Optical recording media, such as optical disks, which carry out recording and deletion of information based on reversible phase transitions of a thin film of Ge-Sb-Te, Ag-In-Sb-Te, etc. caused by an optical beam have a protective film on both sides thereof and a
15 reflective film on one side thereof with the other side being the substrate.

It is known that a dielectric thin film comprising ZnS and SiO_2 formed by sputtering can be used as such a protective film as disclosed in Japanese Patent Application Laid-Open No. 27672/88 and Japanese Patent Publication No. 36164/92. The dielectric thin film comprising ZnS and SiO_2 has been widespread as a protective film because it possesses
20 almost every required characteristic, such that the refractive index falls within a desired range, the thermal conductivity is low, and it does not absorb laser light.

Thin film formation techniques can be adopted to the formation of the ZnS- SiO_2 protective film, and sputtering is a prevailing technique. As to the target of the sputtering, Japanese Patent Application Laid-Open No. 65725/94 proposes a process for producing a
25 sputtering target comprising uniformly mixing high-purity zinc sulfide powder with 20 mol% of high-purity silicon dioxide powder, pressing the mixed powder, and firing the green body.

Primary characteristics required of the sputtering target include resistance to breakage against high sputtering power and generation of no dust (or particulate matter).

If the target breaks, or if dust generates from the target, maintenance of the equipment is often needed, such as a change of the target, which impairs the productivity of optical media, such as optical disks. The increased frequency of changes of the target is also an economical disadvantage.

5 Attention being paid to the crystal structure of zinc sulfide, Japanese Patent Application Laid-Open Nos. 81954/98 and 81960/98 propose methods of preventing generation of dust (or particulate matter) from a sputtering target. The proposed methods, however, failed to sufficiently prevent dust generation or to effectively prevent breakage of the target.

10 Summary of the Invention

 Accordingly, an object of the present invention is to provide a target of sputtering for optical recording protective film formation which has high strength and generates little dust or particulate matter and therefore withstands long-term use and a process for preparing the target.

15 As a result of studies, the present inventors have found it important that the target should have a smooth surface after sputtering in order to prevent dust generation. They have reached the finding that a sputtering target which has high strength and maintains a smooth surface after sputtering under given conditions can be obtained by selecting fine powder as an SiO_2 raw material and pressure sintering mixed powder at high temperature for
20 densification and thus accomplished the above object.

 Based on the above findings, the present invention provides a target of sputtering for optical recording protective film formation which has a surface roughness (Ra) of less than 2.4 after sputtering.

25 The present invention also provides a preferred process for preparing the above-described target of sputtering which comprises mixing ZnS powder and SiO_2 powder and hot pressing the mixed powder, wherein the hot pressed SiO_2 has an average particle size of $2\text{ }\mu\text{m}$ or smaller, and the hot pressing is carried out in an inert gas atmosphere at a temperature of 1200°C or higher.

Brief Description of the Drawings:

Fig. 1 is a scanning electron micrograph (SEM; 500x magnification) of the surface of the target obtained in Example 1 after sputtering.

Fig. 2 is a scanning electron micrograph (SEM; 5000x magnification) of the surface of the target obtained in Example 1 after sputtering.

Fig. 3 is a scanning electron micrograph (SEM; 500x magnification) of the surface of the target obtained in Comparative Example 1 after sputtering.

Fig. 4 is a scanning electron micrograph (SEM; 500x magnification) of the surface of the target obtained in Comparative Example 2 after sputtering.

Fig. 5 is a scanning electron micrograph (SEM; 500x magnification) of the surface of the target obtained in Comparative Example 3 after sputtering.

Fig. 6 is a graph showing the relationship between the average particle size of SiO_2 raw material powder and the surface roughness (Ra) after sputtering and the number of non-defectives.

Fig. 7 is a graph showing the relationship between the surface roughness (Ra) after sputtering and the number of non-defectives.

Detailed Description of the Preferred Embodiments

The present invention will be described hereunder in detail. The target of sputtering for optical recording protective film formation (hereinafter simply referred to as a sputtering target) according to the present invention is made of ZnS and SiO_2 , containing 10 to 30 mol% of SiO_2 based on ZnS .

The sputtering target of the present invention has a surface roughness (Ra) of less than 2.4 after sputtering. With the surface roughness (Ra) exceeding 2.4, nodules will be formed to such a size that can cause dust, and dust, being ascribed to the nodules, easily scatters from the surface of the target on sputtering.

It is preferred for the sputtering target to have a bending strength of 8.5 kgf/mm^2 or more. With a bending strength less than 8.5 kgf/mm^2 , there is a fear that the target may be broken under high sputtering power.

The process of preparation according to the present invention is described below.

The SiO_2 raw material powder should be such that has an average particle size of 2 μm or smaller after sintering. SiO_2 raw material powders that have an average particle size of 2 μm or smaller after sintering include grinds having an average particle size of 2 μm or smaller of an SiO_2 raw material, such as quartz, and agglomerates of SiO_2 powder which can be deflocculated by mixing and grinding. Agglomerates include Aerosil available from Nippon Aerosil Co., Ltd. The ZnS raw material powder used usually has an average particle size of about 2 to 6 μm .

The ZnS powder and the SiO_2 powder are separately mixed with water and ground in a bead mill, etc. into fine powder having a desired particle size. The two slurried powders are mixed, filtered by suction, and dried in the air at 400°C , at which the powder is not oxidized, to remove water, hydrogen sulfide, and sulfur oxides.

The ZnS-SiO_2 mixed powder is hot pressed (i.e., pressure sintering) in an inert gas atmosphere, preferably an argon gas atmosphere, at 1200°C or higher, preferably 1200 to 1300°C . At temperatures below 1200°C , it is difficult to sinter the fine SiO_2 powder, resulting in a failure of obtaining a high-strength sputtering target.

The hot pressing is preferably performed by, for example, elevating the temperature up to 1100°C in vacuo to remove sulfur oxides, then introducing argon gas into the firing furnace, and pressure sintering the powder at 1200°C or higher.

The thus prepared sputtering target maintains a smooth surface even after sputtering under given conditions and has high strength.

The present invention will now be illustrated in greater detail with reference to Example and Test Example.

EXAMPLE 1

ZnS raw material powder (4.33 kg) having an average particle size of 6.3 μm was mixed with 90% by volume of water. The density of the ZnS raw material powder being 4 g/cc, the mixture comprised 1083 cc of ZnS and 9747 cc of water. The mixture was

ground in a bead mill making 3 passes over a total residence time of 4 minutes. The resulting ZnS powder had an average particle size of 2.5 μm .

Separately, 1000 cc of water was poured into 0.67 kg of SiO_2 raw material powder having an average particle size of 2.0 μm (Aerosil OX50), and the mixture was stirred well.

5 The ZnS aqueous slurry and the SiO_2 aqueous slurry were mixed, stirred in a bead mill, and allowed to stand still. The supernatant liquid was removed, and the solid was filtered by suction and dried in the air at 400°C. The resulting mixed powder was preliminarily pressed at 50 kgf/cm², evacuated, and heated up to 1250°C over 80 minutes. At 1100°C argon gas was introduced to one atmosphere. After a temperature of 1250°C
10 was reached, the pressure was increased to 300 kgf/cm² over 5 minutes, under which the powder was maintained for 60 minutes. The pressure was removed over 3 minutes, and the furnace was cooled to obtain a sputtering target.

 The SiO_2 in the target was found to have an average particle size of 2.0 μm or smaller, and the density of the target was 3.67 g/cc. The target was evaluated in terms of
15 surface roughness (Ra) after sputtering, bending strength, and the number of non-defectives.

 The results of evaluation are shown in Table 1 below. The electron scanning micrographs (500x and 5000x magnifications) of the surface of the target after sputtering are shown in Figs. 1 and 2, respectively.

COMPARATIVE EXAMPLE 1

20 A sputtering target was obtained in the same manner as in Example 1, except for using SiO_2 raw material powder having an average particle size of 6.2 μm (quartz grinds).

 The target density was measured, and the target was evaluated in the same manner as in Example 1. The results obtained are shown in Table 1. The scanning electron micrograph (500x magnification) on the surface of the target after sputtering is shown in Fig.
25 3.

COMPARATIVE EXAMPLE 2

ZnS raw material powder (4.33 kg) having an average particle size of 6.3 μm and SiO_2 raw material powder (0.67 kg) having an average particle size of 58.7 μm were mixed in a ball mill. The resulting mixed powder was preliminarily pressed at 50 kgf/cm^2 , evacuated, and heated up to 900°C over 55 minutes, at which the powder was maintained for 80 minutes. After 15 minutes from the time when the temperature reached 900°C, the pressure was increased to 300 kgf/cm^2 over 20 minutes. After the 80 minute hot pressing at 900°C, the pressure was relieved over 2 minutes, followed by cooling the furnace to obtain a sputtering target.

The target density was measured, and the target was evaluated in the same manner as in Example 1. The results obtained are shown in Table 1. The scanning electron micrograph (500x magnification) of the surface of the target after sputtering is shown in Fig. 4.

COMPARATIVE EXAMPLE 3

ZnS raw material powder (4.33 kg) having an average particle size of 6.3 μm and SiO_2 raw material powder (0.67 kg) having an average particle size of 25.1 μm were mixed in a ball mill. The resulting mixed powder was preliminarily pressed at 50 kgf/cm^2 , evacuated, and heated up to 1000°C over 90 minutes, at which the powder was maintained for 120 minutes. After 20 minutes from the time when the temperature reached 1000°C, the pressure was elevated to 300 kgf/cm^2 over 20 minutes. After the 120 minute hot pressing at 1000°C, the pressure was relieved over 2 minutes, followed by cooling the furnace to obtain a sputtering target.

The target density was measured, and the target was evaluated in the same manner as in Example 1. The results obtained are shown in Table 1. The scanning electron micrograph (500x magnification) on the surface of the target after sputtering is shown in Fig. 5.

TABLE 1

	Average Particle Size of Raw Material (μm)		Hot Pressing Temp. ($^{\circ}\text{C}$)	Density of Target (g/cc)	Surface Roughness (R_a) [*] after Sputtering (μm)	Bending Strength (kgf/mm^2)	Number of Non-Defectives ^{**}
	ZnS	SiO_2					
Example 1	2.5	2.0	1250	3.67	0.4	10.1	12000
Compara. Example 1	2.5	6.2	1250	3.60	2.4	8.4	5000
Compara. Example 2	6.3	58.7	900	3.60	6.2	5.1	1000
Compara. Example 3	6.3	25.1	1000	3.21	4.8	1.7	1500

Note: *:Instrument for measuring surface roughness: TENCOR P-10; scanning length: 2000 μm ; scanning speed: 50 $\mu\text{m/sec}$; stylus: 12.5 μm ; stylus pressure: 2.0 mg

** : The number of non-defective sputtering products that were obtained before a change of the shield.

As is apparent from Table 1, the target of Example 1 has a smooth surface after sputtering and a high bending strength as compared with the comparative targets. As a result, the number of non-defectives increased greatly.

TEST EXAMPLE

5 Targets were obtained in the same manner as in Example 1, except for varying the average particle size of the SiO_2 raw material powder. The relationship between the average particle size of the starting SiO_2 and the surface roughness (Ra) after sputtering and the number of non-defectives is shown in Fig. 6. The relationship between the surface roughness (Ra) after sputtering and the number of non-defectives is shown in Fig. 7. As is
10 seen from Figs. 6 and 7, as the average particle size of the SiO_2 raw material powder becomes smaller, the surface roughness decreases, and the number of non-defectives increases.

As described above, the sputtering target for optical recording protective film formation according to the present invention, being excellent in strength, is hardly broken,
15 generates little dust or particulate matter during sputtering and eventually stands up to long-term use. The process of the present invention provides the sputtering target with good productivity.

CLAIMS



1. A target of sputtering for forming a protective film on an optical recording medium, the target having a surface roughness (Ra) of less than 2.4 after sputtering.
2. A process for preparing a target of sputtering for forming a protective film on an optical recording medium, which comprises mixing ZnS powder and SiO₂ powder and hot pressing the mixed powder, wherein the hot pressed SiO₂ has an average particle size of 2 μm or smaller, and the hot pressing is carried out in an inert gas atmosphere at a temperature of 1200°C or higher.

ABSTRACT**TARGET OF SPUTTERING FOR FORMING
PROTECTIVE FILM ON OPTICAL RECORDING
MEDIA AND PROCESS FOR PREPARING THE TARGET**

A sputtering target for optical recording protective film formation comprising ZnS and SiO₂, which has a surface roughness (Ra) of less than 2.4 after sputtering. (Figure 1)

FIG. 1

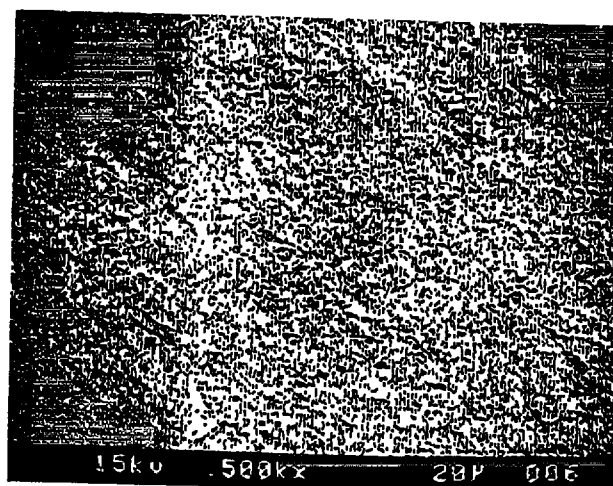


FIG. 2

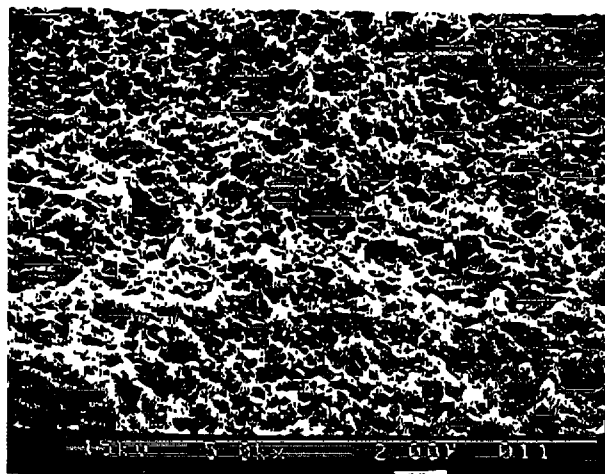


FIG. 3

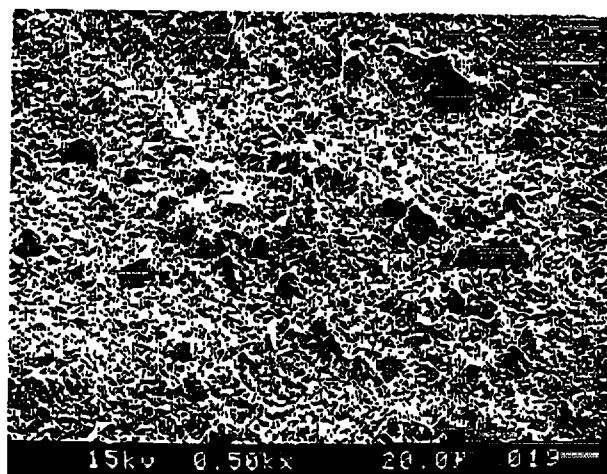


FIG.4

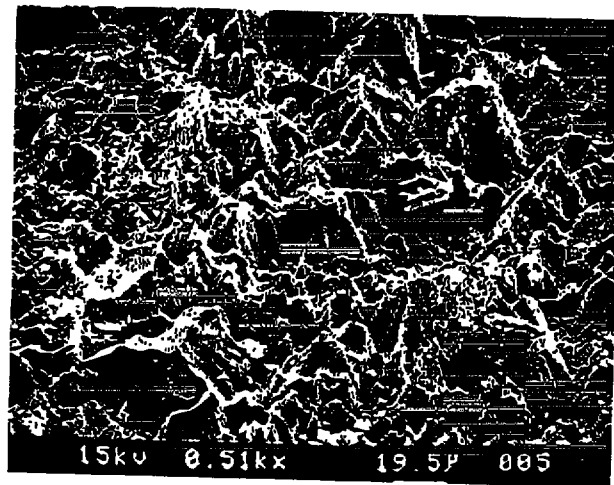


FIG.5

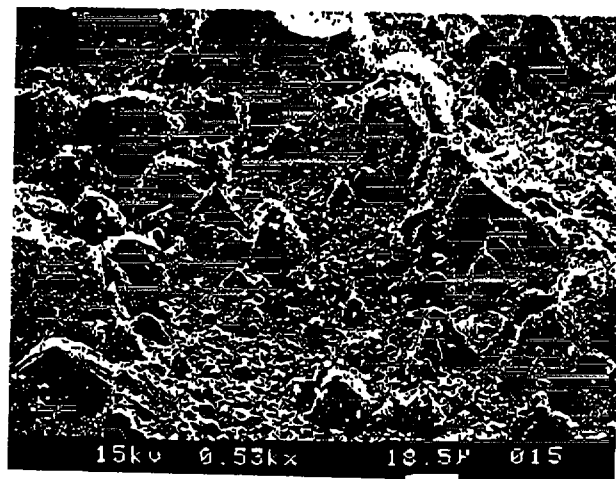


FIG. 6

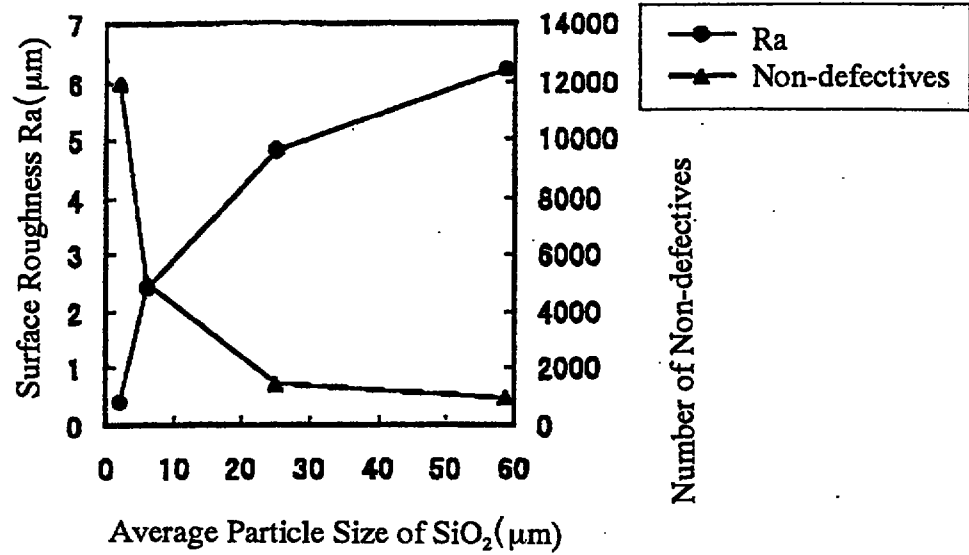


FIG. 7

