MgO-TiO SINTERED COMPACT TARGET AND METHOD FOR PRODUCING SAME

BACKGROUND

[0001] The present invention relates to a magnesium oxide-based target for use in magnetic recording mediums of magnetic disk devices (hard disks) and electrodevices such as tunneling magnetoresistance (TMR) elements, and to a method for producing such a target. The present invention particularly relates to a conductive sintered compact magnesium oxide-based target for use in high density sputtering, and to a method for producing such a target.

[0002] In recent years, pursuant to the miniaturization and higher recording density of magnetic disks, the research and development of magnetic recording mediums are being conducted, and in particular, various improvements in the magnetic layer and base layer have been made. The recording density of hard disks is rapidly increasing year by year, and the current surface density of 600 Gbit/in^2 is expected to reach 1 Tbit/in^2 in the future. When the recording density reaches 1 Tbit/in^2, the size of the recording bit will fall below 10 nm and, in such a case, it is anticipated that the paramagnetism caused by thermal fluctuation will become a problem, and it is further anticipated that the currently used materials; for instance, a material with higher magnetic crystalline anisotropy obtained by adding Pt to a Co-Cr-based alloy, will no longer be sufficient. This is because magnetic grains to stably behave as a ferromagnetic at a size of 10 nm or less need to possess even higher magnetic crystalline anisotropy.

[0003] Based on the reasons described above, an Fe-Pt phase having an $L1_0$ structure is attracting attention as a material for use as an ultrahigh density recording medium. Since an Fe-Pt phase having an $L1_0$ structure yields superior corrosion resistance and oxidation resistance in addition to having high magnetic crystalline anisotropy, it is expected to become a material that can be suitably applied as a magnetic recording medium. Furthermore, in connection with using the Fe-Pt phase as a material for use in an ultrahigh density recording medium, demanded is the development of technology for dispersing the ordered Fe-Pt magnetic grains, in a magnetically isolated state, while densely aligning the orientation thereof as much as possible. In order to provide magnetic anisotropy to an Fe-Pt thin film, the crystal orientation needs to be controlled, and this can be easily realized by selecting a

monocrystal substrate. In order to vertically align the magnetized orientation axis, it has been reported that a magnesium oxide film is suitable as the base layer of the Fe-Pt layer.

[0004] In addition, it is also known that a magnesium oxide film is being used as an insulating layer (tunnel barrier) of TMR elements that are used in magnetic heads (for hard disks) and MRAMs. This kind of magnesium oxide film was conventionally formed via the vacuum deposition method, but recently magnesium oxide films are being produced via the sputtering method in order to facilitate the simplification of the production process and to attain a larger area of the magnesium oxide film. The following patent documents are known as conventional technologies.

[0005] Patent Document 1 below discloses a magnesium oxide target made from a magnesium oxide sintered compact having a magnesium oxide purity of 99.9% higher and a relative density of 99% or more, having a fine structure in which the average grain size is 60 μm or less and round pores having an average grain size of 2 μm or less exist in the crystal grains, and which enables a sputter deposition rate of 1000 Å/min or more. This technology is based on the method of adding and mixing a fine magnesium oxide powder having an average grain size of 100 nm or less to a high purity magnesium oxide powder, molding the mixed powders, and subjecting the obtained sintered compact to primary sintering and secondary sintering.

[0006] Patent Document 2 below proposes a magnesium oxide target made from a magnesium oxide sintered compact having a relative density of 99% more, and capable of attaining a deposition rate of 500 Å/min or more in a sputter deposition that is performed in an Ar atmosphere or an Ar-O_2 mixed atmosphere, wherein the magnesium oxide target is obtained by CIP molding a high density magnesium oxide powder having an average grain size of 0.1 to 2 μm at a pressure of 3 t/cm^2 or more and sintering the obtained sintered compact.

[0007] Patent Document 3 below describes a magnesium oxide target made from a magnesium oxide sintered compact having a magnesium oxide purity of 99.9% or higher and a relative density of 99.0% or more, and capable of attaining a sputter deposition rate of 600 Å/min or more. Patent Document 3 further describes that this magnesium oxide target is obtained by adding and mixing an electromelted magnesium oxide powder and a fine magnesium oxide powder having an average grain size of 100 nm or less to a high purity magnesium oxide powder, molding the mixed powders, and subjecting the obtained sintered compact to primary sintering

and secondary sintering, and that a magnesium oxide film having favorable orientation, crystallinity and film properties can be deposited at a high deposition rate via the sputtering method.

[0008] Patent Document 4 below proposes a target having MgO as its main component and a method of producing such a target, and dispersing La grains, Y grains, and Sc grains in the target having MgO as its main component in order to attain a lower discharge voltage, sputtering resistance properties during discharge, fast discharge responsiveness, and insulation, and enable use as a protective film of a dielectric layer of an A_C-type PDP.

[0009] Patent Document 5 below proposes dispersing LaB_6 particles in a MgO matrix, performing reduction treatment in a reduction gas atmosphere prior to sintering, and performing primary sintering and secondary sintering at a predetermined temperature in order to improve the strength, fracture toughness and thermal shock resistance in a target having MgO as its main component.

[0010] Patent Document 6 below proposes defining the relative density and average crystal grain size to 0.5 to 100 μm in a target having MgO as its main component, and dispersing the rare earth elements of Sc, Y, La, Ce, Gd, Yb, and Nd in a MgO matrix. Patent Document 7 below proposes sintering a MgO compact via the discharge plasma sintering method in order to produce a high density sintered compact.

[0011] Patent Document 8 and Patent Document 9 below propose obtaining a MgO sintered compact in which numerous (111) planes are oriented based on uniaxial pressure sintering by setting the ultimate density to 3.568 g/cm^3 and in order to attain favorable mechanical properties and thermal conductivity, and reduce contamination of the atmosphere caused by gas generation, wherein the MgO raw material powder having a grain size of 1 μm or less is subject to uniaxial pressure sintering, and thereafter subject to heat treatment in an oxygen atmosphere at a temperature of 1273 K or higher. In the foregoing case, MgO is used as the raw material powder, the means for increasing the density is limited to the sintering conditions.

[0012] Patent Document 10 below proposes a target capable of depositing a MgO film on a large scale and in a uniform manner, wherein the average crystal grain size, density, transverse rupture strength, and center line average roughness of the target surface are defined, the grain size of the raw material powder is set to 1 μm or less,

and, after subjecting the target to the granulation process, the target is sintered at a predetermined load and temperature, and subject to surface finishing so that the center line average roughness Ra of the target becomes 1 μm or less. Note that Patent Document 11 describes forming a nonmagnetic seed layer, which is made from any one of the following materials; namely, MgO, NiO, TiO, or Ti carbide having a NaCl-type structure, between a nonmagnetic substrate and a nonmagnetic base layer in a vertical magnetic recording medium.

Prior Art Documents

Patent Documents

[0013] Patent Document 1: Japanese Patent Application Publication No. H10-130827
Patent Document 2: Japanese Patent Application Publication No. H10-130828
Patent Document 3: Japanese Patent Application Publication No. H10-158826
Patent Document 4: Japanese Patent Application Publication No. H10-237636
Patent Document 5: Japanese Patent Application Publication No. H11-6058
Patent Document 6: Japanese Patent Application Publication No. H11-335824
Patent Document 7: Japanese Patent Application Publication No. H11-139862
Patent Document 8: Japanese Patent Application Publication No. 2009-173502
Patent Document 9: Pamphlet of International Publication No. 2009/096384
Patent Document 10: Japanese Patent Application Publication No. 2000-169956
Patent Document 11: Japanese Patent Application Publication No. 2004-213869

SUMMARY

[0014] In recent years, there are increased demands of magnesium oxide films for use in magnetic recording mediums of magnetic disk devices (hard disks) and electrodevices such as tunneling magnetoresistance (TMR) elements. Since magnesium oxide is an insulation material, radio frequency (RF) sputtering is normally adopted. Nevertheless, RF sputtering has inferior productivity since its deposition rate is slow and particles are easily generated, and there is a problem in that RF sputtering deteriorates the quality of the film. Thus, an object of this invention is to provide a high density target having a high deposition rate and which can be subject to direct current (DC) sputtering with low generation of particles, and a method for producing such a target.

[0015] In order to achieve the foregoing object, as a result of intense study, the

present inventors discovered that a conductive sintered compact, which can be subject to DC sputtering, can be obtained by attaining a composite oxide target in which conductive titanium oxide TiO, which has the same NaCl-type crystal structure and a similar lattice constant as magnesium oxide MgO, is mixed with magnesium oxide MgO; and the functions as a base film or the like are not impaired, since the obtained film also possesses the equivalent crystal structure as magnesium oxide.

[0016] Based on the foregoing discovery, the present invention provides:

1) A MgO-TiO sintered compact comprising TiO in an amount of 25 to 90 mol%, and remainder being MgO and unavoidable impurities;

2) The MgO-TiO sintered compact according to 1) above, wherein relative density is 95% or more;

3) The MgO-TiO sintered compact according to 1) or 2) above, wherein bulk resistivity is 10 Ω•cm or less;

4) The MgO-TiO sintered compact according to any one of 1) to 3) above, wherein two phases of a TiO phase and a MgO phase exist, and a number of regions in which a longest diameter of the MgO phase is 50 μm or more is 10 or less per 1 mm^2; and

5) A method of producing a MgO-TiO sintered compact for a sputtering target which contains 10 mol% or more and 90 mol% or less of TiO in MgO, wherein raw material powders made from a MgO powder having an average grain size of 10 μm or less and a TiO powder having an average grain size of 50 μm or less are mixed, and the mixed powders are hot-pressed at a temperature of 1250 to 1450°C and a pressure of 200 kgf/cm^2 or more in order to prepare the MgO-TiO sintered compact.

[0017] The present invention can provide a magnesium oxide-based sintered compact having high density and low bulk resistivity. When this sintered compact is used as a sputtering target, the target can be used for deposition via DC sputtering and, therefore, since the deposition rate can be dramatically improved and stable sputtering is enabled, a superior effect is yielded in that the amount of particles that are generated is small. Moreover, since an expensive RF power source for RF sputtering is not required, the present invention yields a superior effect of being able to reduce costs for the capital investments.

BRIEF DESCRIPTION OF THE DRAWINGS

[0018] Fig. 1 is an image of the structure of the target of Example 2 observed using a laser microscope.

Fig. 2 is an image of the structure of the target of Example 2 observed using a laser microscope (image in which the image of Fig. 1 was scaled down by approximately 1/5).

DETAILED DESCRIPTION

[0019] A major characteristic of the MgO-TiO sintered compact of the present invention is that TiO is added to MgO. Since a conductive sintered compact made from MgO-TiO can be obtained by adding TiO possessing conductivity, a sputtering target prepared by using this sintered compact can be subject to DC sputtering, and the amount of particles that are generated during sputtering can also be reduced.

[0020] As described above, the present invention is unique in that, by adding TiO possessing conductivity, the sintered compact is given conductivity to enable DC sputtering, but what is further important is that the TiO has the same NaCl-type crystal structure as MgO and adopts a lattice constant of a value that is similar to that of MgO, and is of the same oxide as MgO but does not react with MgO to generate an intermediate compound. Consequently, the film formed by sputtering yields a superior effect of not losing its characteristics in comparison to a conventional film made independently from magnesium oxide.

Moreover, as the conductive material that can be applied in the present invention, TiN, TiC, CrN, NbN, NbC, TaN, TaC, ZrN, ZrC, VN, VC and the like may be used in addition to TiO. When based only from the perspective of the lattice constant, TiC, VC, WC, and TiN are promising, but the carbides or nitrides thereof contain numerous oxygen impurities in the raw material powder and may become decomposed, reduce the oxygen of MgO, or generate an intermediate compound with MgO when mixed with MgO and sintered, and the characteristics (lattice constant and the like) that are inherent in Mg or "TiC, VC, WC, TiN" may become lost.

[0021] In the MgO-TiO sintered compact of the present invention, the TiO content is 25 mol% or more and 90 mol% or less, and preferably 35 mol% or more and 70 mol% or less. When the TiO content is less than 25 mol%, it is difficult to obtain the bulk resistance that enables DC sputtering, and this is undesirable. Meanwhile, when the TiO content exceeds 90 mol%, the characteristics of the formed film approach

those of pure TiO, and the intended characteristics cannot be obtained, and this is also undesirable.

Note that the present invention also covers cases of adding other materials so as long as the addition is within a range in which DC sputtering is enabled and the characteristics of the film are not considerably changed.

[0022] Moreover, in the MgO-TiO sintered compact of the present invention, the relative density is preferably 95% or more, and more preferably 98% or more. When this kind of high density sintered compact is used as a target for sputtering, it is possible to reduce the amount of particles that are generated during sputtering.

[0023] Moreover, in the MgO-TiO sintered compact of the present invention, the bulk resistivity is preferably 10 Ω•cm or less, and more preferably 0.01 Ω•cm or less. When a sintered compact having a low bulk resistance value is used as a target for sputtering, more stable DC sputtering is enabled. Since it is thereby possible to increase the deposition rate in comparison to conventional RF sputtering, the productivity can be improved.

Note that, even if the bulk resistivity exceeds the foregoing range, it should be easy to understand that such a case is also covered by the present invention so as long as DC sputtering is enabled.

[0024] Moreover, in the MgO-TiO sintered compact of the present invention, the two phases of a TiO phase and a MgO phase exist, and the number of regions in which the longest diameter of the MgO phase is 50 μm or more is preferably 10 regions or less per 1 mm^2, and the number of regions in which the longest diameter of the MgO phase is 30 μm or more is more preferably 25 regions or less per 1 mm^2. The TiO phase is desirably dispersed by being connected in a net-like shape.

[0025] The present invention is a sintered compact in which MgO and TiO having considerably different conductivity coexist, but when a coarse MgO phase exists, an abnormal discharge tends to occur with such coarse MgO phase as the origin. The occurrence of an abnormal discharge with a coarse MgO phase as the starting point can be inhibited by reducing the regions of such a coarse MgO phase as much as possible, and the amount of particles can also be reduced. Note that the term "longest diameter of the MgO phase" refers to the maximum length of the grains forming the MgO phase on the polished surface of a sample taken from a part of the target.

[0026] The MgO-TiO sintered compact of the present invention can be prepared according to the following method.

Foremost, a MgO powder and a TiO powder are prepared as the raw material. Preferably, the MgO powder has an average grain size of 10 μm or less, and the TiO powder has an average grain size of 50 μm or less. When the grain size of the powders exceeds the foregoing range, it is undesirable since uniform mixture becomes difficult, and segregation and coarsening of crystal grains will occur. While a finer grain size of the raw material powder is desirable, since it is difficult to pulverize TiO, the average grain size is preferably set to 1 μm or more from the perspective of productivity.

Subsequently, the raw material powders are weighed to achieve a predetermined molar ratio, and mixed and pulverized using a well-known means such as a ball mill.

[0027] The thus obtained mixed powder is molded and sintered via the hot-pressing method in a vacuum atmosphere or an inert gas atmosphere. Moreover, the plasma discharge sintering method and various other pressure sintering methods may be used in addition to the hot-pressing method. In particular, the hot isostatic sintering method is effective in improving the density of the sintered compact. The holding temperature during sintering is preferably within the temperature range of 1250 to 1450°C. Moreover, the holding pressure during sintering is preferably within the pressure range of 200 kgf/cm^2 or more.

[0028] Moreover, in the present invention, a sputtering target can be prepared by processing the thus obtained sintered compact into an intended shape via grinding or the like. Since the sputtering target produced as described above can be subject to DC sputtering, the deposition rate can be dramatically increased and the productivity can be considerably improved. In addition, since the amount of particles that are generated during sputtering can be reduced, the present invention yields a superior effect of being able to improve the yield during deposition.

[Examples]

[0029] The present invention is now explained based on the following Examples and Comparative Examples. Note that these Examples are merely exemplifications, and the present invention is not in any way limited by these Examples. In other words, the present invention is limited only by the scope of claims, and various modifications other than the following Examples included in the present invention are also covered by this invention.

[0030] (Example 1)

As raw material powders, a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%), and a TiO powder having an average grain size of 30 μm and a purity of 3N (99.9%) were prepared. These raw material powders were blended to achieve the composition ratio shown in Table 1.

Subsequently, the weighed powders were sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium under an Ar atmosphere, and mixed and pulverized by rotating the ball mill pot for 20 hours or longer so that both powders become uniformly dispersed.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1400°C, and a pressure of 250 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

[0031] As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 98%. Here, the term "relative density" refers to the value obtained by dividing the measured density of the target by the calculated density (also known as the theoretical density). The calculated density is the density when it is assumed that the constituents of the target coexist without diffusing or reacting with each other, and the formula thereof is as follows: calculated density = Σ (molar weight of constituents × molar ratio of constituents) / Σ (molar weight of constituents × molar ratio of constituents / theoretical density of constituents). Note that 3.585 g/cm^3 was adopted as the theoretical density of MgO, and 4.93 g/cm^3 was adopted as the theoretical density of TiO in all of the following Examples and Comparative Examples.

Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the bulk resistance was 0.01 Ω•cm. Moreover, as a result of grinding the cross section of the sintered compact and observing the central part with a laser microscope, the two phases of a MgO phase and a TiO phase were observed, and the regions in which the longest diameter of the MgO phase is 50 μm or more and 30 μm or more were 5 regions/mm^2 and 15 regions/mm^2, respectively.

[0032] In addition, the sintered compact was polished into a target shape using a grinding machine to prepare a disk-shaped target. The prepared target was mounted

on a DC sputtering machine and sputtered. The sputtering conditions were as follows; namely, sputter power of 0.5 kW and Ar gas pressure of 5 Pa, and a film was deposited on a silicon substrate for 30 seconds. Subsequently, the number of particles that became adhered to the substrate was measured using a particle counter. The number of particles in the foregoing case was 120 particles.

[0033] [Table 1]

	Composition (mol%) MgO:TiO	Density (%)	Bulk Resistivity	Number of regions in which longest diameter of MgO phase is 50 μm or more (regions/mm^2)	Number of regions in which longest diameter of MgO phase is 30 μm or more (regions/mm^2)	Number of Particles
Example 1	75:25	98	0.01	5	15	120
Example 2	50:50	98	0.003	3	5	51
Example 3	30:70	99.5	0.002	0	5	46
Example 4	10:90	99.5	0.0005	0	0	22
Comparative Example 1	100:0	99	-	-	-	DC sputtering not possible
Comparative Example 2	90:10	96	-	13	35	DC sputtering not possible
Comparative Example 3	50:50	97	0.007	25	53	2000
Comparative Example 4	30:70	99.5	0.002	15	41	500

[0034] (Example 2)

As raw material powders, a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%), and a TiO powder having an average grain size of 20 μm and a purity of 3N (99.9%) were prepared. These raw material powders were blended to achieve the composition ratio shown in Table 1.

Subsequently, the weighed powders were sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium under an Ar atmosphere, and mixed and pulverized by rotating the ball mill pot for 20 hours or longer so that both powders become uniformly dispersed.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1400°C, and a pressure of 300 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

[0035] As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 98%. Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the bulk resistance was 0.003 Ω•cm. Moreover, the cross section of the sintered compact was ground and the central part was observed with a laser microscope. The results are shown in Fig. 1. As shown in Fig. 1, the two phases of a MgO phase (dark gray portion) and a TiO phase (light gray portion) were observed. Moreover, the regions in which the longest diameter of the MgO phase is 50 μm or more and 30 μm or more were respectively one region and two regions as shown in Fig. 2, and when these image regions are converted into areas per 1 mm^2, the regions were 3 regions/mm^2 and 5 regions/mm^2, respectively. Note that, in the other Examples and Comparative Examples (excluding Comparative Example 1) also, it was confirmed that the sintered compact is formed from the two phases of a MgO phase and a TiO phase based on the structure image having the same magnification as Fig. 1, and the number of MgO phases in which the longest diameter is 50 μm or more and 30 μm or more was counted in the image region having the same magnification as Fig. 2, then converted into areas per 1 mm^2, and used as the number of regions per unit area.

[0036] In addition, the sintered compact was polished into a target shape using a grinding machine to prepare a disk-shaped target. The prepared target was mounted on a DC sputtering machine and sputtered. The sputtering conditions were as follows; namely, sputter power of 0.5 kW and Ar gas pressure of 5 Pa, and a film was deposited on a silicon substrate for 30 seconds. Subsequently, the number of particles that became adhered to the substrate was measured using a particle counter. The number of particles in the foregoing case was 51 particles.

[0037] (Example 3)

As raw material powders, a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%), and a TiO powder having an average grain size of 30 μm and a purity of 3N (99.9%) were prepared. These raw material powders were blended to achieve the composition ratio shown in Table 1.

Subsequently, the weighed powders were sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium under an Ar atmosphere, and mixed and pulverized by rotating the ball mill pot for 20 hours or longer so that both powders become uniformly dispersed.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1400°C, and a pressure of 250 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

[0038] As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 99.5%. Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the bulk resistance was 0.002 Ω•cm. Moreover, as a result of grinding the cross section of the sintered compact and observing the central part with a laser microscope, the two phases of a MgO phase and a TiO phase were observed, and the regions in which the longest diameter of the MgO phase is 50 μm or more and 30 μm or more were 0 regions/mm^2 and 5 regions/mm^2, respectively.

[0039] In addition, the sintered compact was polished into a target shape using a grinding machine to prepare a disk-shaped target. The prepared target was mounted on a DC sputtering machine and sputtered. The sputtering conditions were as follows; namely, sputter power of 0.5 kW and Ar gas pressure of 5 Pa, and a film was deposited on a silicon substrate for 30 seconds. Subsequently, the number of particles that became adhered to the substrate was measured using a particle counter. The number of particles in the foregoing case was 46 particles.

[0040] (Example 4)

As raw material powders, a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%), and a TiO powder having an average grain size of 30 μm and a purity of 3N (99.9%) were prepared. These raw material powders were blended to achieve the composition ratio shown in Table 1.

Subsequently, the weighed powders were sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium under an Ar atmosphere, and mixed and pulverized by rotating the ball mill pot for 10 hours or longer so that both powders become uniformly dispersed.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1400°C, and a pressure of 250 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

[0041] As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 99.5%. Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the bulk resistance was 0.0005 Ω•cm. Moreover, as a result of grinding the cross section of the sintered compact and observing the central part with a laser microscope, the two phases of a MgO phase and a TiO phase were observed, and the regions in which the longest diameter of the MgO phase is 50 μm or more and 30 μm or more were 0 regions/mm^2 and 0 regions/mm^2, respectively.

[0042] In addition, the sintered compact was polished into a target shape using a grinding machine to prepare a disk-shaped target. The prepared target was mounted on a DC sputtering machine and sputtered. The sputtering conditions were as follows; namely, sputter power of 0.5 kW and Ar gas pressure of 5 Pa, and a film was deposited on a silicon substrate for 30 seconds. Subsequently, the number of particles that became adhered to the substrate was measured using a particle counter. The number of particles in the foregoing case was 22 particles.

[0043] (Comparative Example 1)

As raw material powders, only a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%) was prepared. This powder was sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium, and pulverized by rotating the ball mill pot for 10 hours.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1500°C, and a pressure of 300 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 99%. Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the resistance value was high and the bulk resistance could not be measured.

The obtained sintered compact was machined into a target shape using a lathe to prepare a disk-shaped target. The prepared target was mounted on a DC sputtering machine and sputtered, but DC sputtering could not be performed.

[0044] (Comparative Example 2)

As raw material powders, a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%), and a TiO powder having an average grain size of 25 μm and a purity of 3N (99.9%) were prepared. These raw material powders were blended to achieve the composition ratio shown in Table 1.

Subsequently, the weighed powders were sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium under an Ar atmosphere, and mixed and pulverized by rotating the ball mill pot for 20 hours or longer so that both powders become uniformly dispersed.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1400°C, and a pressure of 300 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

[0045] As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 96%. Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the resistance value was high and the bulk resistance could not be measured. Moreover, as a result of grinding the cross section of the sintered compact and observing the central part with a laser microscope, the two phases of a MgO phase and a TiO phase were observed, and the regions in which the longest diameter of the MgO phase is 50 μm or more and 30 μm or more were 13 regions/mm^2 and 35 regions/mm^2, respectively.

[0046] In addition, the sintered compact was polished into a target shape using a grinding machine to prepare a disk-shaped target. The prepared target was mounted on a DC sputtering machine and sputtered, but DC sputtering could not be performed.

[0047] (Comparative Example 3)

As raw material powders, a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%), and a TiO powder having an average grain size of 100 μm and a purity of 3N (99.9%) were prepared. These raw material powders were blended to achieve the composition ratio shown in Table 1.

Subsequently, the weighed powders were sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium under an Ar atmosphere, and mixed and pulverized by rotating the ball mill pot for 5 hours or longer so that both powders become uniformly dispersed.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1400°C, and a pressure of 300 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

[0048] As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 97%. Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the bulk resistance was 0.007 Ω•cm. Moreover, as a result of grinding the cross section of the sintered compact and observing the central part with a laser microscope, the two phases of a MgO phase and a TiO phase were observed, and the regions in which the longest diameter of the MgO phase is 50 μm or more and 30 μm or more were 25 regions/mm^2 and 53 regions/mm^2, respectively.

[0049] In addition, the sintered compact was polished into a target shape using a grinding machine to prepare a disk-shaped target. The prepared target was mounted on a DC sputtering machine and sputtered. The sputtering conditions were as follows; namely, sputter power of 0.5 kW and Ar gas pressure of 5 Pa, and a film was deposited on a silicon substrate for 30 seconds. Subsequently, the number of particles that became adhered to the substrate was measured using a particle counter. The number of particles in the foregoing case was 2000 particles.

[0050] (Comparative Example 4)

As raw material powders, a MgO powder having an average grain size of 1 μm and a purity of 4N (99.99%), and a TiO powder having an average grain size of 100 μm and a purity of 3N (99.9%) were prepared. These raw material powders were blended to achieve the composition ratio shown in Table 1.

Subsequently, the weighed powders were sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as the grinding medium under an Ar atmosphere, and mixed and pulverized by rotating the ball mill pot for 5 hours or longer so that both powders become uniformly dispersed.

Subsequently, the powder removed from the pot was filled in a graphite die having a diameter of 180 mm, and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a holding temperature of 1400°C, and a pressure of 300 kgf/cm^2 was applied from the time that the temperature increase was started to the end of the holding time.

[0051] As a result of measuring the density of the thus prepared sintered compact based on the Archimedes method, the relative density was 99.5%. Moreover, as a result of measuring the bulk resistance of the sintered compact based on the four-terminal method, the bulk resistance was 0.002 Ω•cm. Moreover, as a result of grinding the cross section of the sintered compact and observing the central part with a laser microscope, the two phases of a MgO phase and a TiO phase were observed, and the regions in which the longest diameter of the MgO phase is 50 μm or more and 30 μm or more were 15 regions/mm^2 and 41 regions/mm^2, respectively.

[0052] In addition, the sintered compact was polished into a target shape using a grinding machine to prepare a disk-shaped target. The prepared target was mounted on a DC sputtering machine and sputtered. The sputtering conditions were as follows; namely, sputter power of 0.5 kW and Ar gas pressure of 5 Pa, and a film was deposited on a silicon substrate for 30 seconds. Subsequently, the number of particles that became adhered to the substrate was measured using a particle counter. The number of particles in the foregoing case was 500 particles.

[0053] Since the MgO-TiO sintered compact of the present invention can be subject to DC sputtering, the deposition rate can be dramatically increased in comparison to subjecting a conventional MgO sintered compact to RF sputtering, and a significant effect of being able to improve the productivity is yielded. Moreover, since DC sputtering can be realized through use of an inexpensive DC power source, existing equipment can be used as is, and capital investment costs can be reduced.

Accordingly, the MgO-TiO sintered compact of the present invention is effective as a magnesium oxide-based sputtering target for use in forming thin films of magnetic recording mediums for use in magnetic disk devices and electrodevices such as tunneling magnetoresistance (TMR) elements. Moreover, the present invention can also be used in new fields such as static electricity removal and heat resistant members as a conductive ceramic material that could not be realized with conventional insulation MgO.

CLAIMS

1. A MgO-TiO sintered compact comprising TiO in an amount of 25 to 90 mol%, and remainder being MgO and unavoidable impurities.

2. The MgO-TiO sintered compact according to claim 1, wherein relative density is 95% or more.

3. The MgO-TiO sintered compact according to claim 1 or claim 2, wherein bulk resistivity is 10 Ω•cm or less.

4. The MgO-TiO sintered compact according to any one of claims 1 to 3, wherein two phases of a TiO phase and a MgO phase exist, and a number of regions in which a longest diameter of the MgO phase is 50 μm or more is 10 or less per 1 mm^2.

5. A method of producing a MgO-TiO sintered compact for a sputtering target which contains 10 mol% or more and 90 mol% or less of TiO in MgO, wherein raw material powders made from a MgO powder having an average grain size of 10 μm or less and a TiO powder having an average grain size of 50 μm or less are mixed, and the mixed powders are hot-pressed at a temperature of 1250 to 1450°C and a pressure of 200 kgf/cm^2 or more in order to prepare the MgO-TiO sintered compact.