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DESCRIPTION

TECHNICAL FIELD

[0001] This invention relates to a method for selecting a magneto-optical ceramic material that can be used in the construction of magneto-optical devices (for example, Faraday rotators) in optical isolators.

BACKGROUND ART

[0002] With the advance of laser processing machines, magneto-optical devices utilizing the interaction of light and magnetism have recently become of much interest. One of these devices is an isolator, which functions to inhibit the phenomenon that if the light oscillated from a laser source is reflected by the optical system in its path and is returned to the light source, then it disturbs the light oscillated from the laser source, to cause an unstable oscillation state. Accordingly, on use, the optical isolator is arranged between a laser source and an optical member so as to take advantage of the function.

[0003] The optical isolator comprises three parts, a Faraday rotator, a polarizer arranged on the light-input side of the Faraday rotator, and an analyzer arranged on the light-output side of the Faraday rotator. The optical isolator utilizes the nature, commonly known as the Faraday effect, that when light enters the Faraday rotator under the condition where a magnetic field is applied to the Faraday rotator in a direction parallel to the light traveling direction, the plane of polarization is rotated in the Faraday rotator. Specifically, the light of the incident light having the same plane of polarization as that of the polarizer is transmitted by the polarizer and enters the Faraday rotator. The light is rotated by +45 degrees relative to the light traveling direction in the Faraday rotator, and then emerges therefrom.

[0004] By contrast, when the return light entering the Faraday rotator in an opposite direction to the incident direction first passes through the analyzer, only a component of the light having the same plane of polarization as that of the analyzer is transmitted by the analyzer and enters the Faraday rotator. In the Faraday rotator, the plane of polarization of the return light is further rotated by +45 degrees in addition to the initial +45 degrees. Since the plane of polarization of the return light is at a right angle of +90 degrees with respect to the polarizer, the return light is not transmitted by the polarizer.

[0005] It is necessary that the material to be used for the Faraday rotator of the optical isolator mentioned above have a significant Faraday effect and a high transmittance at the wavelength at which it is used.

[0006] Also, if a polarized component different from the incident light is generated in the output

light, this different polarized component is transmitted by the polarizer, indicating insufficient blockage of the return light.

[0007] For the evaluation of the generation of the different polarized component, polarized light of 0 to 90 degrees enters a material used as the Faraday rotator, output light is transmitted by the polarizer into a photodetector, and the intensity of light is measured by the photodetector. From the maximum intensity ($I_{\max}$) and minimum intensity ($I_{\min}$), an extinction ratio (S) is computed according to the following equation.

$$S = -10 \log (I_{\min}/I_{\max}) \quad (\text{unit: dB})$$

While higher values of extinction ratio are desirable, an extinction ratio of at least 30 dB is generally required.

[0008] JP-A 2010-285299 discloses a single crystal oxide of $(\text{Tb}_x\text{Re}_{1-x})_2\text{O}_3$ (wherein $0.4 \leq x \leq 1.0$) and transparent oxide ceramics as the material having a high Verdet constant.

[0009] As compared with the single crystal oxide, the transparent oxide ceramics are inexpensive and industrially promising because the reaction temperature can be kept low so that large-scale production in a simple plant is possible.

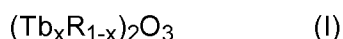
[0010] In JP 4033451, for example, a rare earth oxide of the general formula: R_2O_3 wherein R is a rare earth element is free of birefringence since its crystal structure is cubic. It is described that a sintered body having a high degree of transparency can be produced if pores and impurity segregates are completely removed.

[0011] Also, JP-A H05-330913 discloses that addition of sintering aids is effective for removing pores. Further, JP 2638669 discloses removal of pores by hot isostatic pressing (HIP) and re-sintering.

[0012] On the other hand, if heat treatment in the presence of sintering aids is continued for a long time, the sintering aids or the like segregates at grain boundaries. Sometimes, a difference arises between the refractive index of the main phase of crystal grains and the refractive index at the grain boundary.

[0013] If there is a difference in refractive index between the main phase and the grain boundary as mentioned above, the polarization state of transmitted light is changed, specifically the extinction ratio becomes lower. An optical isolator manufactured using such material has a poor degree of optical separation.

[0014] JP 2010-285299 is directed to an oxide that contains the oxide represented by Formula (I) as the main component thereof, that has a Verdet constant at a wavelength of $1.06 \mu\text{m}$ of at least $0.18 \text{ min}/(\text{Oe} \cdot \text{cm})$, and that has a transmittance at a wavelength of $1.06 \mu\text{m}$ and for an optical length of 3 mm of at least 70%,



wherein X is $0.4 \leq X \leq 1.0$; R includes at least one element selected from the group consisting of scandium, yttrium, lanthanum, europium, gadolinium, ytterbium, holmium and lutetium.

[0015] WO 2006/285299 is directed to a light-transparent material that is high in space filling ratio and, less likely to cause light scattering, and has excellent light transmittance. After the synthetic of particles by liquid phase synthesis, the solution is replaced without drying the particles, and a solution comprising a composition of a grain boundary phase or a liquid containing a precipitate of at least one member selected from Al_2O_3 , yttrium oxide, silicon oxide, yttrium-silicon composite oxide, aluminum-silicon composite oxide, or compounds having a garnet structure with a melting point below the melting point of the above particles is introduced.

SUMMARY OF INVENTION

Technical Problem

[0016] An object of the invention is to provide a method for selecting a magneto-optical ceramic material having a satisfactory polarization state and a high extinction ratio.

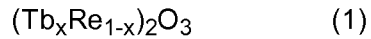
Solution to Problem

[0017] The inventors made extensive investigations to overcome the above problem. In connection with the process of preparing a ceramic material by using the oxide of terbium which is a paramagnetic element with a high Verdet constant and an oxide of rare earth element (scandium, yttrium, lanthanum, europium, gadolinium, ytterbium, holmium or lutetium) as main components, adding one or more sintering aids thereto, mixing, molding, calcining, sintering in vacuum, HIP treating, and annealing, ceramic materials were prepared while the conditions of each step including time, temperature and atmosphere were changed. Examining the relationship of the difference in refractive index between main phase and grain boundary of the resulting ceramic particles to the extinction ratio, the inventors have found that a highly homogeneous ceramic material substantially free of segregation of sintering aids at the grain boundary and having an improved extinction ratio is obtained if the difference in refractive index is less than 0.004.

[0018] Accordingly, the invention provides a method for selecting a magneto-optical ceramic material as defined below.

1. [1] A method for selecting a magneto-optical ceramic material having a high extinction ratio comprising the step of selecting an oxide ceramic material wherein the difference

between the refractive index at the grain boundary and the refractive index of the main phase of the oxide ceramic crystal at 25 °C is not more than 0.004 as a magneto-optical ceramic material having an extinction ratio of at least 30 dB, from oxide ceramic materials based on an oxide having the formula (1):



wherein Re is at least one element selected from the group consisting of scandium, yttrium, lanthanum, europium, gadolinium, ytterbium, holmium and lutetium, and $0.4 \leq x \leq 1.0$,

wherein the oxide ceramic materials based on an oxide having the above formula (1) are prepared by using a terbium oxide and an oxide of at least one rare earth element selected from the group consisting of yttrium, scandium, lanthanum, europium, gadolinium, ytterbium, holmium and lutetium as main components, adding one or more sintering aids thereto, mixing, molding, calcining, sintering in vacuum, and hot isostatic pressing,

characterized in that the difference between the refractive index at the grain boundary and the refractive index of the main phase of the oxide ceramic crystal at 25°C is measured by steps of polishing end faces of a oxide ceramic material to mirror finish, resting the oxide ceramic material on a movable stage, directing measurement light to a microscopic spot of the measurement surface of the oxide ceramic material via an objective lens while moving the oxide ceramic material, detecting the intensity of the reflected light from the measurement surface by a photodetector receiving the reflected light continuously, and determining the difference between the refractive index at the grain boundary and the refractive index of the main phase of the oxide ceramic crystal from a change of the photodetector signal.

2. [2] The method of [1], wherein the relationship of a change of refractive index to a change of photodetector signal is precorrected by attaching a material having a known refractive index to the sample, performing mirror finish polishing perpendicular to the attachment interface and measuring the sample in this state.
3. [3] The method of [1] or [2], wherein the extinction ratio is evaluated by directing polarized light of 0 degrees to 90 degrees to the oxide ceramic material, directing output light to a photodetector via a polarizer, measuring the intensity of light in the photodetector, and calculating an extinction ratio (S) according to the following equation from the maximum intensity (I_{max}) and minimum intensity (I_{min}),

$$S = -10 \log (I_{\min}/I_{\max}) \quad (\text{unit: dB}).$$

Advantageous Effects of Invention

[0019] The invention ensures to provide a magneto-optical ceramic material having a

satisfactory polarization state and high extinction ratio, suited for use in optical isolator Faraday rotators or the like.

BRIEF DESCRIPTION OF DRAWINGS

[0020]

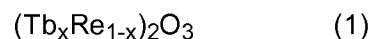
FIG. 1 schematically illustrates a system for measuring the refractive index of grain boundary and main phase.

FIG. 2 is a schematic diagram showing one exemplary relationship of positional information to reflected light intensity.

FIG. 3 is a graph showing extinction ratio versus refractive index difference.

DESCRIPTION OF EMBODIMENTS

[0021] The magneto-optical ceramic material is selected from oxide ceramic materials based on an oxide having the formula (1) :



wherein Re is at least one element selected from the group consisting of scandium, yttrium, lanthanum, europium, gadolinium, ytterbium, holmium and lutetium, and $0.4 \leq x \leq 1.0$, the oxide ceramic material preferably having a Verdet constant of at least 0.18 min/Oe-cm at wavelength 1.065 μm wherein the difference between the refractive index at the grain boundary and the refractive index of the main phase of the oxide ceramic crystal at 25°C is not more than 0.004, and the extinction ratio is γ at least 30 dB.

[0022] In formula (1), x is at least 0.4, preferably 0.5 to 0.9, and more preferably 0.5 to 0.7. Re is selected from the above-listed elements, with Y and Gd being preferred.

[0023] The oxide ceramic material contains an oxide, fluoride, nitride or another compound of titanium, zirconium, hafnium, calcium or the like as sintering aid and may optionally contain 0.0001 to 0.01 part by weight of an alkaline earth metal oxide such as magnesium oxide, strontium oxide or barium oxide per 100 parts by weight of the oxide of formula (1).

[0024] The oxide ceramic material is prepared by the method described in JP-A 2010-285299, specifically by providing the oxide of terbium which is a paramagnetic element with a high Verdet constant and an oxide of rare earth element (scandium, yttrium, lanthanum, europium, gadolinium, ytterbium, holmium or lutetium) as main components, adding thereto one or more

sintering aids, for example, the sintering aids disclosed in JP-A H05-330913, specifically an oxide, fluoride, nitride or another compound of titanium, zirconium, hafnium, calcium or the like, mixing, molding, calcining, sintering in vacuum, and HIP treating according to the procedure commonly used in the prior art.

[0025] Of the oxide ceramic materials thus obtained, those in which the difference between the refractive index at the grain boundary and the refractive index of the main phase of the oxide ceramic crystal is not more than 0.004 are selected and adopted as the homogeneous material having a high extinction ratio.

[0026] Based on the principle that the intensity of reflected light varies with refractive index, the difference in refractive index between the grain boundary and the main phase may be determined by the following method shown in FIG. 1.

[0027] Once a ceramic material (or sample) 1 in test at its end faces is polished to mirror finish, it is rested on a moving stage 2. The stage 2 is attached to a ball screw 4 coupled to a motor 3 so that the stage 2 is movable. A pedestal is depicted at 5.

[0028] Measurement light from a light source 6 is directed to the sample 1 obliquely from above. The measurement light is expanded in a beam expander 7, reflected by a mirror 8, and directed to the sample 1 via an objective lens 9, whereby measurement of a microscopic spot is possible. As the measurement wavelength becomes shorter, measurement of a finer spot is possible. A photodetector (or power meter) 10 receives the reflected light for detecting the intensity of reflected light.

[0029] While the test sample is moved, signals from the photodetector are monitored. When the refractive index changes at the grain boundary, the signal from the photodetector changes. The reflectivity R at the interface between the sample and air is expressed as $R = (n-1)^2/(n+1)^2$ wherein n is the refractive index of the sample.

[0030] Then the refractive index is determined from a change of the photodetector signal.

[0031] It is noted that FIG. 2 shows one exemplary relationship of the intensity of reflected light from the power meter to the positional information from the motor.

[0032] Since the relationship of a change of refractive index to a change of photodetector signal varies with an optical measurement system, this relationship can be corrected by attaching a material having a known refractive index to the sample, performing mirror finish polishing perpendicular to the attachment interface and measuring the sample in this state.

[0033] Also the difference in refractive index may be qualitatively evaluated by slicing the test sample and visually observing the slice under a microscope.

[0034] Separately, an approximate estimate is obtainable by a visual comparison of a test

sample with the standard sample whose refractive index difference has been measured with the reflected light.

EXAMPLES

[0035] A mixture was obtained by combining a terbium oxide powder and a Y_2O_3 or Gd_2O_3 powder having a different particle size in a molar ratio (40-70% : 60-30%), adding 0.5 wt% of ZrO_2 , HfO_2 or TiO_2 as sintering aid thereto, further adding effective amounts of ethyl cellulose as dispersant and polyvinyl alcohol as binder, and milling them in a pot mill. The mixture was spray dried, yielding granules having a size of several tens of micrometer. The granules were molded in a mold as primary shaping and subjected to CIP as secondary shaping into a compact. The compact was calcined in air at 400-1,000°C and fired in a predetermined atmosphere at 1,600-1,800°C. The fired body was further subjected to HIP and optionally annealed. In this way, fifteen ceramic materials (size: diameter 6 mm, length 10 mm) were obtained. Each of the thus obtained ceramic materials was measured for a difference between the refractive index at the grain boundary and the refractive index of the main phase and an extinction ratio. The results are shown in the diagram.

[0036] It is noted that the refractive index is determined by using Ellipsometer as a refractive index meter, and measuring a Brewster's angle at 25°C. The extinction ratio is evaluated by directing polarized light of 0 degree to 90 degrees to a material to be used as a Faraday rotator, directing output light to a photodetector via a polarizer, measuring the intensity of light in the photodetector, and calculating an extinction ratio (S) according to the following equation from the maximum intensity (I_{max}) and minimum intensity (I_{min}).

$$S = -10 \log (I_{min}/I_{max}) \quad \{\text{unit: dB}\}$$

[0037] The results are shown in FIG. 3.

[0038] It has been demonstrated that the extinction ratio is at least 30 dB as long as the refractive index difference is 0.004 or less. As the refractive index difference is reduced, the extinction ratio is improved.

Reference Signs List

[0039]

- 1
ceramic material (sample)
- 2
moving stage

- 3 motor
- 4 ball screw
- 5 pedestal
- 6 light source
- 7 beam expander
- 8 mirror
- 9 objective lens
- 10 photodetector

REFERENCES CITED IN THE DESCRIPTION

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- [JP2010285299A \[0008\] \[0014\] \[0024\]](#)
- [JP4033451B \[0010\]](#)
- [JPH05330913A \[0011\] \[0024\]](#)
- [JP2638669B \[0011\]](#)
- [WO2006285299A \[0015\]](#)

KRAV

1. Fremgangsmåde til udvælgelse af et magneto-optisk keramisk materiale, som har et højt ekstinktionsforhold, hvor fremgangsmåden omfatter trinnet at vælge et oxid-keramisk materiale, hvor forskellen mellem refraktionsindekset ved korngrænsen og refraktionsindekset af hovedfasen af det oxid-keramiske materiale ved 25 °C ikke er mere end 0.004 som et magneto-optisk keramisk materiale, der har et ekstinktionsforhold på mindst 30 dB, ud fra oxid-keramiske materialer baseret på en oxid med følgende formel (1):



hvor Re er mindst ét grundstof udvalgt fra gruppen, der består af scandium, yttrium, lanthan,

europium, gadolinium, ytterbium, holmium and lutetium, og $0.4 \leq x \leq 1.0$, hvor det oxid-keramiske materiale, der har en ovenstående formel (1), er fremstillet ved anvendelse af en terbiumoxid og en oxid af mindst én sjælden jordart udvalgt fra gruppen, der består af yttrium, scandium, lanthan, europium, gadolinium, ytterbium, holmium and lutetium som primær komponent, tilsætte dertil af én eller flere sintring hjælpemidler, sammenblanding, støbning,

kalcinerung, sintring i vakuum, og varm isostatisk presning,

kendetegnet ved, at forskellen mellem refraktionsindekset ved korngrænsen og refraktionsindekset af hovedfasen af den oxid-keramiske krystal ved 25 °C er målt ved trinnene at blankpolere endeflader af et oxid-keramisk materiale, at anbringe det oxid-keramiske materiale på et bevægeligt objektbord, rette målelys mod et mikroskopisk punkt af måleoverfladen af det oxid-keramiske materiale via et objektiv mens det oxid-keramiske materiale bevæges, at detektere intensiteten af det reflekterede lys fra måleoverfladen ved hjælp af en fotodetektor, som kontinuert modtager det reflekterede lys, og at bestemme forskellen mellem refraktionsindekset ved korngrænsen og refraktionsindekset af hovedfasen af den oxid-keramiske krystal ud fra en ændring i fotodetektorsignalet.

2. Fremgangsmåde ifølge krav 1, hvor forholdet mellem en ændring af refraktionsindeks til en ændring af fotodetektorsignalet er kompenseret ved at knytte et materiale med et kendt refraktionsindeks til prøven, at udføre blankpolering vinkelret til tilknytningsgrænsefladen, og at måle prøven i denne tilstand.

3. Fremgangsmåde ifølge krav 1 eller 2, hvor ekstinktionsforholdet er evalueret ved at rette polariseret lys ved 0 grader til 90 grader mod det oxid-keramiske materiale, at rette udgangsllys mod en fotodetektor via en polarisator, at måle lysintensiteten i fotodetektoren, og at beregne et ekstinktionsforhold (S) ifølge den følgende ligning fra maksimumintensiteten (I_{max}) og minimumintensiteten (I_{min}),

$$S = -10 \log(I_{\text{min}}/I_{\text{max}}) \quad (\text{enhed: dB}).$$

DRAWINGS

FIG.1

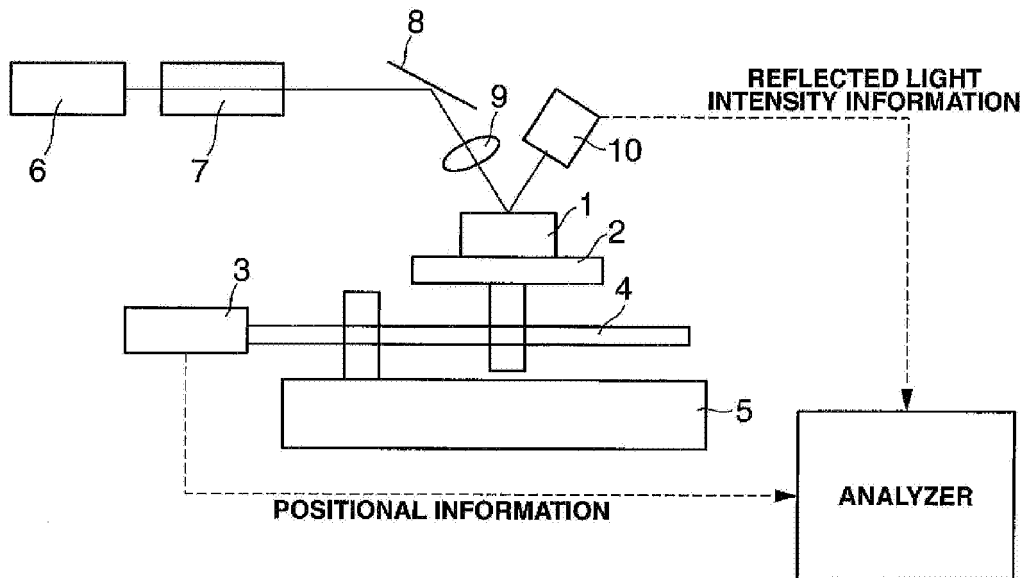


FIG.2

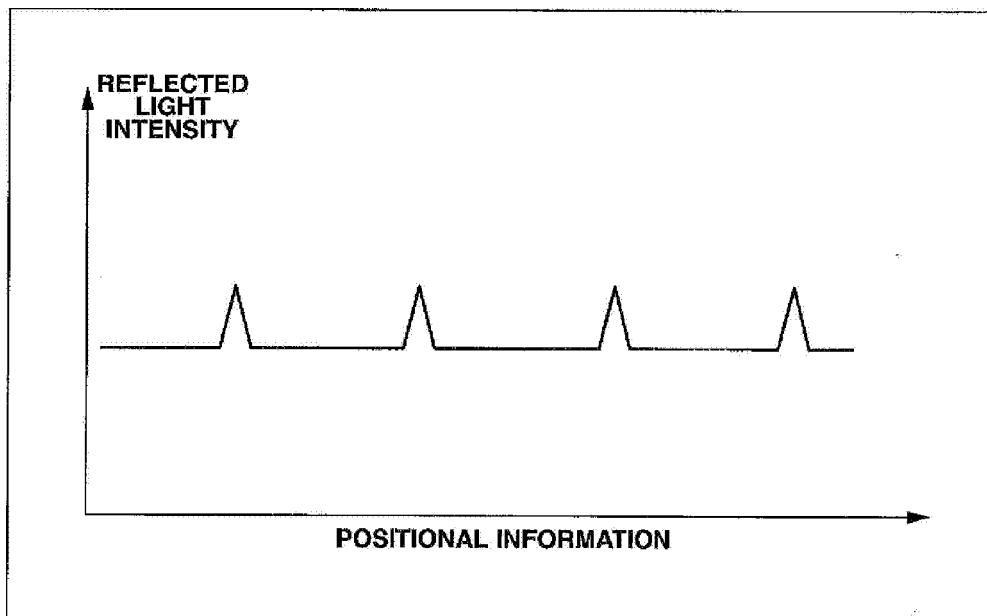


FIG.3