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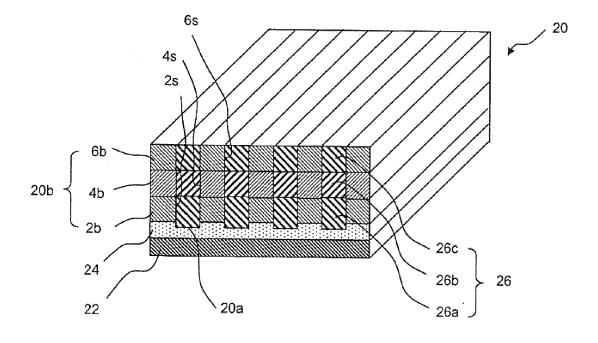
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- (54) Diffraction grating for x-ray talbot interferometer, method of manufacturing the same, and x-ray talbot interferometer
- (57) A diffraction grating (20) for an X-ray Talbot interferometer includes: a plurality of ridge-like X-ray absorbing portions (20b) made of a metal and formed on a substrate (22) along one direction at predetermined intervals; and a resin (26) interposed in a groove portion (20a) between neighboring X-ray absorbing portions of

the plurality of ridge-like X-ray absorbing portions. The plurality of ridge-like X-ray absorbing portions are each formed of at least two unit metal layers (2b, 4b, and 6b) laminated perpendicularly to a surface of the substrate. The at least two unit metal layers are formed through cutting of a metal film.

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#### Description

[0001] The present invention relates to a diffraction grating for an X-ray Talbot interferometer, a method of manufacturing the same, and an X-ray Talbot interferometer.

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[0002] There is known a Talbot effect, in which a diffraction grating is used and self images of the diffraction grating are formed at specific distances from the diffraction grating when light from a coherent light source passes through the diffraction grating. In recent years, there has been developed an X-ray Talbot interferometer that uses this Talbot effect so as to detect a phase shift of a transmitted X-ray. An image obtained by the phase shift of an X-ray using the Talbot effect is advantageous in having higher contrast particularly in a substance having a small atomic number than a conventional image obtained by an absorbed amount of a transmitted X-ray.

[0003] As such an X-ray Talbot interferometer, there is known a structure illustrated in FIG. 10. The X-ray Talbot interferometer 1000 includes a first diffraction grating 1010, a second diffraction grating 1020, and an X-ray image detector 30 (see International Patent WO 2004/58070 A). As illustrated in FIGS. 11A and 11B, the first diffraction grating 1010 and the second diffraction grating 1020 have grooves 1010a and 1020a formed in a metal plate at predetermined intervals in one direction. The groove transmits the X-ray, while a ridge portion 1010b between neighboring grooves transmits the X-ray after shifting the phase by  $\pi/2$ , and a ridge portion 1020b blocks (absorbs) the X-ray. As a material of the diffraction grating, gold (Au) having high X-ray absorbing power is usually used.

[0004] In this X-ray Talbot interferometer, when the Xray is irradiated from an X-ray source to the first diffraction grating via a sample, the X-ray transmitted through the groove portion 1010a and the X-ray transmitted and diffracted through the ridge portion 1010b interfere with each other. Then, self images of the first diffraction grating 1010 are formed at positions of integral multiples of a Talbot distance  $d2/2\lambda$  of the first diffraction grating 1010 (d represents a period of the diffraction grating, and  $\lambda$ represents a wavelength of the X-ray) as the Talbot effect. The self image has a distortion due to a sample 4, and the distortion has information of the sample. The second diffraction grating 1020 is disposed at a position where the self image of the first diffraction grating 1010 is formed. Then, a distribution of the X-ray transmitted through the second diffraction grating 1020 has moire fringes because the self image of the first diffraction grating is overlaid. Therefore, this X-ray distribution is detected by the X-ray image detector, and image analysis is performed so that an image of the sample 4 is obtained. In order to improve image contrast, it is preferred that the groove portion 1020a of the second diffraction grating 1020 have a high X-ray transmittance, while the ridge portion 1020b have a low X-ray transmittance. Therefore, it is preferred that the second diffraction grating 1020 be

an amplitude diffraction grating having a larger thickness than the first diffraction grating 1010.

[0005] Here, in order to generate the Talbot effect, it is necessary that the ridge portions (X-ray absorbing portions) of the diffraction grating have a period securing coherency of the X-ray. Therefore, the period of the ridge portion is required to be approximately 10 µm or smaller. Further, in a phase diffraction grating, the contrast of the self image becomes highest when a phase shift amount becomes  $\pi/2$ . Therefore, in order to realize this, it is necessary to set a thickness of the ridge portion (depth of the groove) at approximately 1 to 10 µm, and fine machining and manufacturing technique are required.

[0006] On the other hand, in order to function as the amplitude diffraction grating, it is preferred that the groove portion 1020a of the diffraction grating have a high X-ray transmittance and the ridge portion 1020b have a low X-ray transmittance. Therefore, it is required to form the groove to have a large depth of approximately 10 to 100  $\mu m$  even when gold is used. Therefore, an aspect ratio expressed by (depth of groove)/(width of groove) of the diffraction grating becomes very large, and hence production of the diffraction grating becomes difficult.

25 [0007] Therefore, there is disclosed a technique of manufacturing the diffraction grating for the X-ray Talbot interferometer, in which a deep groove is formed in a resin by X-ray lithography using an X-ray mask, and the X-ray absorbing portion is formed in the groove by an electroforming method (see Japanese Patent Application Laid-open No. 2006-259264).

[0008] In addition, there is disclosed a technique including the steps of patterning and removing a photosensitive resin on a surface of a silicon substrate by a lithography method, etching the silicon substrate, from which the photosensitive resin is removed, by an ICP plasma etching method so as to form a slit groove, then depositing an insulator material in the slit groove, further etching the remaining photosensitive resin and the silicon substrate by the ICP plasma etching method so as to form a second slit groove, and forming an X-ray absorbing metal portion in the second slit groove by the electroforming method (see Japanese Patent Application Laidopen No. 2009-42528).

[0009] However, in the technique described in Japanese Patent Application Laid-open No. 2006-259264 or Japanese Patent Application Laid-open No. 2009-42528, the electroforming is performed in the fine groove (slit), and hence it is more difficult to perform the electroforming securely as the aspect ratio of the groove becomes higher. In addition, when a resist resin is used for forming the groove having a high aspect ratio, there is a problem that it is difficult to manufacture the diffraction grating with high dimensional accuracy because the resin is a soft insulator, which may be deformed so that neighboring ridge portions come into contact with each other (sticking). Further, in order to form the groove having a high aspect ratio, it is necessary to use synchrotron radiation

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light so that exposure is performed by the X-ray having high linearity, which largely increases manufacturing cost.

**[0010]** Therefore, an object of the present invention is to provide a diffraction grating for an X-ray Talbot interferometer, a method of manufacturing the same, and an X-ray Talbot interferometer, the method enabling easy and highly accurate manufacturing of a diffraction grating having grooves with a high aspect ratio.

[0011] According to an exemplary embodiment of the present invention, there is provided a diffraction grating for an X-ray Talbot interferometer, including: a plurality of ridge-like X-ray absorbing portions made of a metal and formed on a substrate along one direction at predetermined intervals; and a resin interposed in a groove portion between neighboring X-ray absorbing portions of the plurality of ridge-like X-ray absorbing portions. The plurality of ridge-like X-ray absorbing portions are each formed of at least two unit metal layers laminated perpendicularly to a surface of the substrate. The at least two unit metal layers are formed through cutting of a metal film. Roughness of a side wall of the groove portion of each of the at least two unit metal layers has an arithmetical mean height Ra of 0.1 µm or smaller, as defined in JIS B0601.

**[0012]** With this structure, the groove portion is formed in the metal film by the cutting tool, and hence it is possible to obtain the diffraction grating having a high dimensional accuracy of the side wall of the unit metal layer, as well as a high dimensional accuracy of the side wall of the X-ray absorbing portion, compared with the conventional technique of manufacturing the diffraction grating by performing electroforming in the fine groove (slit) formed by using a resist resin.

[0013] Further, in order to form the groove portion having a high aspect ratio expressed by (depth of groove portion)/(width of groove portion) through one cutting operation, it is necessary to use an elongated monocrystal diamond cutting tool. The cutting tool may be easily broken, and dimensional accuracy of a shape of the side wall of the groove portion may be deteriorated. Therefore, the depth of the groove portion formed through one cutting operation is set to a value that is not extremely large (the aspect ratio is set to a value that is not extremely high), and the unit metal layers formed through individual cutting operations are laminated. Thus, the aspect ratio of the finally obtained X-ray absorbing portion (groove portion) is high, and break of the cutting tool is reduced. [0014] It is preferred that each of the plurality of ridgelike X-ray absorbing portions contain gold as a main component and be formed of one of crystals each having an average grain diameter of 0.1 µm or smaller and amorphous gold.

**[0015]** In this way, the finished surface (side wall) obtained through the cutting process is smooth without a burr, and the characteristic of the diffraction grating is improved.

[0016] According to an exemplary embodiment of the

present invention, there is provided a method of manufacturing a diffraction grating for an X-ray Talbot interferometer, the method including: forming an initial metal film on a substrate; cutting the initial metal film along one direction at predetermined intervals so as to form a plurality of first groove portions and to form a ridge-like first unit metal layer between neighboring groove portions of the plurality of first groove portions; filling a resin in each of the plurality of first groove portions; exposing an upper surface of the ridge-like first unit metal layer through one of grinding and ashing of a surface of the resin; laminating a second metal film on at least the upper surface of the ridge-like first unit metal layer; and cutting the second metal film along a side wall of the first ridge-like unit metal layer so as to form a plurality of second groove portions and to form a ridge-like second unit metal layer between neighboring groove portions of the plurality of second groove portions. The laminating the second metal film and the cutting the second metal film are repeated at least one time in the stated order.

[0017] With this structure, the groove portion is formed in the metal film by the cutting tool, and hence it is possible to obtain the diffraction grating having a high dimensional accuracy of the side wall of the unit metal layer, as well as a high dimensional accuracy of the side wall of the X-ray absorbing portion, compared with the conventional technique of manufacturing the diffraction grating by performing electroforming in the fine groove (slit) formed by using a resist resin.

[0018] Further, in order to form the groove portion having a high aspect ratio expressed by (depth of groove portion)/(width of groove portion) through one cutting operation, it is necessary to use an elongated monocrystal diamond cutting tool. The cutting tool may be easily broken, and dimensional accuracy of a shape of the side wall of the groove portion may be deteriorated. Therefore, the depth of the groove portion formed through one cutting operation is set to a value that is not extremely large (the aspect ratio is set to a value that is not extremely high), and the unit metal layers formed through individual cutting operations are laminated. Thus, the aspect ratio of the finally obtained X-ray absorbing portion (ridge portion) is high, and break of the cutting tool is reduced.

**[0019]** In addition, when the metal film is cut so that each unit metal layer is formed, the resin supports the unit metal layer on the lower side so that inclination or deformation of the unit metal layer is prevented.

**[0020]** It is preferred that the cutting the metal film include adjusting a cutting position of the metal film with reference to an alignment mark disposed on a surface of the substrate.

**[0021]** In this way, after the substrate is transferred in each process step (for example, the metal layer exposing step), the substrate can be carried back to the cutting machine at a precise position, and hence the metal film can be cut precisely along the side wall of the unit metal layer.

[0022] It is preferred that the method further include

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forming an intermediate layer between the substrate and the initial metal film, the intermediate layer having a smaller hardness than the substrate and containing an element having a smaller effective atomic number than an element contained in the initial metal film.

**[0023]** The substrate is usually harder than the initial metal film, and hence the cutting tool is worn out when cutting the substrate. However, with the formation of the intermediate layer between the initial metal film and the substrate, the metal film can be securely cut by an amount corresponding to the thickness thereof so that a deep groove portion can be formed. In addition, through cutting a part of the intermediate layer that is softer than the substrate, the cutting tool is prevented from being worn out. In addition, the intermediate layer has a smaller effective atomic number than the metal film, and hence blocking and a phase change of the X-ray by the intermediate layer are smaller than those by the unit metal layer, which does not affect image quality of an obtained phase image by the diffraction grating.

**[0024]** It is preferred that the resin include at least one of polyimide and a para-xylene polymer because such a resin is superior in mechanical strength and in addition, has high durability with respect to an X-ray in phase imaging.

**[0025]** In at least one of the filling a resin and filling a second resin, it is preferred to fill polyimide, as the resin, in at least one of the each of the plurality of first groove portions and each of the plurality of second groove portions by a vacuum injection method because polyimide can flow into a deep portion of the groove portion.

**[0026]** In at least one of the filling a resin and filling a second resin, it is preferred to fill a para-xylene polymer, as the resin, in at least one of the each of the plurality of first groove portions and each of the plurality of second groove portions by a vacuum vapor deposition method because the para-xylene polymer can flow into the deep portion of the groove portion.

**[0027]** In the cutting the initial metal film and the cutting the metal film, it is preferred to use a cutting tool having a cutting blade made of monocrystal diamond because monocrystal diamond has high hardness and enables precise machining of the groove.

**[0028]** An X-ray Talbot interferometer according to an exemplary embodiment of the present invention uses the above-mentioned diffraction grating for an X-ray Talbot interferometer.

**[0029]** According to the present invention, the diffraction grating for an X-ray Talbot interferometer having grooves with a high aspect ratio can be manufactured easily with high accuracy.

**[0030]** Embodiments of the present invention will now be described by way of further example only and with reference to the accompanying drawings, in which:

FIG. 1 is a diagram illustrating a schematic structure of an X-ray Talbot interferometer according to an embodiment of the present invention;

FIGS. 2A and 2B illustrate cross sections of a first diffraction grating and a second diffraction grating, which are taken along an x direction;

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FIG. 3 is a perspective view illustrating a structure of a diffraction grating for the X-ray Talbot interferometer according to the embodiment of the present invention;

FIG. 4 is a perspective view illustrating a tool main body to which a monocrystal diamond cutting tool is mounted;

FIG. 5 is a perspective view illustrating the monocrystal diamond cutting tool;

FIG. 6 is a diagram illustrating a method of forming each unit metal layer using the monocrystal diamond cutting tool;

FIGS. 7A to 7D are process charts illustrating an example of a method of manufacturing the diffraction grating for the X-ray Talbot interferometer;

FIGS. 8A to 8E are process charts succeeding FIGS. 7A to 7D;

FIG. 9 is a plan view of an alignment mark disposed on a surface of a substrate;

FIG. 10 is a diagram illustrating a schematic structure of a conventional X-ray Talbot interferometer; and FIGS. 11A and 11B illustrate cross sections of a first diffraction grating and a second diffraction grating of the conventional X-ray Talbot interferometer, which are taken along an x direction.

[0031] Hereinafter, an embodiment of the present invention is described. FIG. 1 is a diagram illustrating a schematic structure of an X-ray Talbot interferometer 100 according to the embodiment of the present invention. The X-ray Talbot interferometer 100 includes an X-ray source 2, a first diffraction grating 10, a second diffraction grating 20, and an X-ray image detector 30. The first diffraction grating 10 and the second diffraction grating 20 are disposed in parallel with a predetermined distance therebetween in a z direction. The X-ray source 2 is disposed to be opposed to the first diffraction grating 10 along the z direction. In addition, the X-ray image detector 30 is disposed to be opposed to the second diffraction grating 20 along the z direction. Further, a sample 4 to be observed is disposed between the first diffraction grating 10 and the X-ray source 2 along the z direction.

[0032] The first diffraction grating 10 and the second diffraction grating 20 have a plurality of groove portions 10a and 20a that are formed to extend along one direction parallel to a plane thereof (y direction in FIG. 1) and be spaced apart from one another in a predetermined period (see the cross section of the groove portions of FIGS. 2A and 2B). The groove portions 10a and 20a transmit the X-ray, while a strip-like ridge portion 10b between the neighboring groove portions 10a transmits the X-ray after shifting the phase by  $\pi/2$ , and a ridge portion 20b blocks (absorbs) the X-ray. The groove portions 10a and 20a, and the ridge portions 10b and 20b extend in the Y direction of FIG. 1. As a material of the diffraction grating,

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it is preferred to use gold having high X-ray absorbing power. Note that, in this embodiment, a width (interval) of the ridge portion 10b and a width (interval) of the groove portion 10a are the same, and a width (interval) of the ridge portion 20b and a width (interval) of the groove portion 20a are the same.

[0033] In the X-ray Talbot interferometer 100, when the X-ray is irradiated from the X-ray source 2 to the first diffraction grating 10 via the sample 4, the X-ray transmitted through the groove portion 10a interferes with the X-ray transmitted and diffracted through the ridge portion 10b. Then, the self image of the first diffraction grating is formed at a position separated by the Talbot distance. In other words, the first diffraction grating 10 constitutes the phase diffraction grating that performs phase modulation on the irradiated X-ray. Here, in order to generate the Talbot effect, it is necessary to adjust the period d of the ridge portion of the first diffraction grating 10 (see FIG. 2A) so as to secure coherency of the X-ray irradiated from the X-ray source 2.

[0034] In addition, the X-ray image detector 30 to the rear of the second diffraction grating 20 detects, as an image contrast, the X-ray transmitted through the groove portion 20a of the second diffraction grating 20 among the self images of the first diffraction grating 10. In order to improve the image contrast, it is preferred that the groove portion 20a of the second diffraction grating 20 have a high X-ray transmittance and the ridge portion 20b have a low X-ray transmittance. Therefore, it is preferred that the second diffraction grating 20 be an amplitude diffraction grating having a larger thickness than the first diffraction grating 10.

**[0035]** Here, the sample 4 is disposed in front of the first diffraction grating 10 and the irradiated X-ray passes through the sample 4 in a slightly different light path, and hence the self image has a distortion due to the sample 4 in accordance with the phase difference at this time. Therefore, when the second diffraction grating 20 is overlaid at a position of the self image, a moire fringe is generated in a Talbot interference image (image contrast), which is detected by the X-ray image detector 30. An amount of modulation of the generated moire fringe by the sample 4 is proportional to an angle of bending of the irradiated X-ray by the sample 4. Therefore, through analysis of the moire fringe, the sample 4 and an internal structure thereof can be measured.

[0036] Note that, a fringe scan method as one of analyzing methods of the moire fringe focuses attention on the fact that a phase of the moire fringe is changed by shifting the first diffraction grating 10 and the second diffraction grating 20 relatively in the X direction. In other words, the phase of the moire fringe is changed so as to obtain a plurality of Talbot interference images, and then the plurality of Talbot interference images are combined by an integral process. Thus, the phase image (the sample 4 and the internal structure thereof) can be obtained. [0037] In addition, it is also possible to obtain a tomographic image (CT image) of the sample 4 by rotating

the sample 4 so as to obtain images from many irradiation directions, and by combining the images.

[0038] Note that, the X-ray Talbot interferometer 100 of the present invention includes a Talbot-Lau interferometer in which a multi-slit is disposed between the X-ray source 2 and the sample 4. When the multi-slit is not used, it is necessary to use a microfocus X-ray source as the X-ray source 2. In contrast, a usual X-ray source can be used in the Talbot-Lau interferometer.

[0039] The X-ray has a short wavelength, and hence, in order to secure coherency, the period of the ridge portion of the first diffraction grating 10 and the second diffraction grating 20 is required to be approximately 10 µm or smaller. Further, in the phase diffraction grating, the contrast of the self image becomes highest when a phase shift amount becomes  $\pi/2$ . Therefore, in order to realize this, it is necessary to set the thickness of the ridge portion (depth of the groove) at approximately 1 to 10 µm, and fine machining and manufacturing techniques are required. For instance, when the ridge portion of each diffraction grating is formed of gold, it is necessary to set the thickness of the ridge portion to approximately 1 to 3  $\mu m$ . When the ridge portion is formed of copper, it is necessary to set the thickness of the ridge portion to approximately 3 to 10 µm.

[0040] On the other hand, in order to function as the amplitude diffraction grating, it is necessary to set a high X-ray transmittance of the groove portion of the diffraction grating and to set a low X-ray transmittance of the ridge portion. Therefore, it is required to set the thickness of the ridge portion (depth of the groove) to a large value of approximately 10 to 100  $\mu m$  even when gold is used. Therefore, the aspect ratio expressed by (depth of groove)/(width of groove) of the diffraction grating becomes quite as large as three or larger (ten or larger in some cases).

**[0041]** For this reason, it is necessary to form the X-ray absorbing portion (ridge portion) finely and to form a shape of the side wall thereof to be sharp (in other words, clearly defined such that a roughness of the side wall of the groove and a curvature radius of a cut corner portion formed by the side wall and the bottom surface are fine). The inventors of the present invention found that a fine X-ray absorbing portion (groove portion) having the side wall with a sharp shape can be formed by cutting a metal film using, for example, a monocrystal diamond cutting tool that has a high hardness and enables precise machining of the groove.

**[0042]** FIG. 3 illustrates a structure of the diffraction grating 20 for the X-ray Talbot interferometer according to the embodiment of the present invention. The diffraction grating 20 for the X-ray Talbot interferometer is the second diffraction grating (amplitude diffraction grating) 20 described above with reference to FIG. 1, and the aspect ratio expressed by (depth of groove)/(width of groove) is preferably three or higher. Note that, the present invention can be applied also to the first diffraction grating 10 as the phase diffraction grating, but is

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preferably applied at least to the above-mentioned amplitude diffraction grating having a high aspect ratio. Boundaries of unit metal layers 2b, 4b, and 6b can be discriminated (measured) by observing a state of roughness of the side wall of the groove portion of each unit metal layer or a positional deviation of each unit metal layer through cross section observation.

[0043] The diffraction grating 20 for the X-ray Talbot interferometer includes a substrate 22, a plurality of ridge-like X-ray absorbing portions 20b which are made of a metal and formed on the substrate 22 along one direction at predetermined intervals, and a resin 26 interposed in the groove portion 20a between the neighboring X-ray absorbing portions. The X-ray absorbing portion 20b is formed by cutting a metal film (described later). A roughness of a side wall 20s of the X-ray absorbing portion 20b and a curvature radius of a cut corner portion formed by the side wall 20s and the bottom of the groove portion are 0.1 µm or smaller each. More particularly, the roughness of the side wall 20s has an arithmetical mean height Ra of 0.1  $\mu m$  or smaller, as defined in JIS B0601. Preferably, the plurality of ridge-like X-ray absorbing portions 20b contain gold as a main component and are formed of one of crystals each having an average grain diameter of 0.1  $\mu m$  or smaller and amorphous gold. Note that, the roughness of the side wall, squareness, and straightness mostly depend on a shape of the cutting tool. Accuracy of a moving mechanism of a superfine nano machine described later is higher than roughness of the surface of the cutting tool.

**[0044]** In addition, an intermediate layer 24 is interposed between the X-ray absorbing portion 20b and the substrate 22.

[0045] It is preferred that the substrate 22 be made of a material having at least one main component selected from the group consisting of carbon, silicon, and aluminum, for example, in order to increase the X-ray transmittance. As a specific example of composition of the substrate 22, there is an amorphous carbon wafer or a silicon wafer, a silicon nitride membrane or a silicon carbide membrane, a 3000-series aluminum sheet, an aluminum-magnesium alloy sheet, or the like, for example. [0046] With the use of the above-mentioned material as the substrate 22, the X-ray transmittance can be enhanced so that a good diffraction characteristic can be obtained. In addition, the X-ray absorption and phase shift amounts depend on a thickness of the metal film, and hence it is preferred that a cutting residue of the metal film does not exist in the bottom of the groove portion. Here, as to a movement of the cutting machine when machining a large number of grooves, the cutting tool is moved only in the direction along the groove in one groove cutting operation, and a constant direction of the depth of the groove is maintained so that the machining can be performed at high speed. In this case, in order to eliminate the cutting residue of the metal film, it is preferred that the substrate including the metal film and the intermediate layer be flat. However, both film thickness

and unevenness of the substrate when the substrate is mounted to the cutting machine have a variation of a few microns or smaller each. Therefore, the flatness of the substrate including the metal film and the intermediate layer is set to be preferably 10  $\mu m$  or smaller, and further the intermediate layer is formed into a thickness equal to or larger than this flatness. Thus, it is possible to prevent the cutting residue of the metal film from remaining, and to prevent the cutting tool from being worn out by cutting the substrate.

[0047] The intermediate layer 24 contains an element having a lower hardness (Vickers' hardness) than that of the substrate and a smaller effective atomic number than that of the metal film (the metal film serving as the X-ray absorbing portion 20b). This element may be a light element that is soft and easily transmits the X-ray, such as a metal (for example, aluminum). The intermediate layer 24 may be a resin. The intermediate layer 24 prevents the cutting tool from cutting the substrate 22 through excessive cutting when the metal film on the substrate 22 is cut so as to form the X-ray absorbing portion 20b. The substrate 22 is usually harder than the metal film, and hence the cutting tool is worn out by cutting the substrate 22. However, with the formation of the intermediate layer 24 between the metal film and the substrate 22, the metal film can be securely cut by an amount corresponding to the thickness thereof to form the deep groove portion. In addition, a part of the intermediate layer 24 softer than the substrate 22 is cut to prevent the cutting tool from being worn out. Note that, the intermediate layer itself has small X-ray absorption and phase change, and hence there is no problem even in a case where a part of the intermediate layer 24 is cut when forming the Xray absorbing portion 20b.

**[0048]** The intermediate layer 24 may be an aluminum layer or a multilayer film including an aluminum layer. However, the intermediate layer 24 is not an essential structure.

[0049] In addition, the intermediate layer 24 has a smaller effective atomic number than the metal film (unit metal layers 2b to 6b), and hence blocking and a phase change of the X-ray by the intermediate layer 24 are smaller than those by the unit metal layer, which does not affect image quality of an obtained phase image by the diffraction grating.

**[0050]** The X-ray absorbing portion 20b can be formed by cutting a metal film containing gold as a main component, for example, a pure gold plating film, or a film of gold-nickel alloy plating or gold-nickel-tungsten alloy plating containing 90% or higher weight ratio of gold, which efficiently absorbs the X-ray so that a characteristic of the diffraction grating is improved. Note that, the metal film includes not only a pure metal but also an alloy as described above.

**[0051]** The resin 26 is filled in the groove after the groove portion is formed so as to hold the X-ray absorbing portion 20b and prevent the X-ray absorbing portion 20b from being inclined or deformed. In particular, it is pre-

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ferred to use at least one of polyimide and a para-xylene polymer as the resin 26 because such a material is superior in mechanical strength and has high durability with respect to X-ray irradiation, particularly in phase imaging. As the para-xylene polymer, there is a polymer in which a part or a whole of hydrogen of the benzene ring of the para-xylene polymer or para-xylene is substituted with chlorine, or a polymer in which  $\alpha$ -hydrogen atoms of paraxylene are substituted with fluorine, and parylene N, parylene C, parylene D, and parylene HT (all of which are registered trademarks) are commercially available. [0052] Next, with reference to FIGS. 4 to 6, the cutting tool, which has a cutting blade made of monocrystal diamond and is suitable for cutting the metal film to form the X-ray absorbing portion 20b, is described. The monocrystal diamond has high hardness and enables precise machining of the groove.

**[0053]** FIG. 4 illustrates a tool main body (cutter) 400 to which a monocrystal diamond cutting tool 200 is mounted. The monocrystal diamond cutting tool 200 is mounted to a tip of a base metal 300 having a substantially trapezoidal shape so as to constitute the tool main body 400. The cutting blade of the monocrystal diamond cutting tool 200 (see FIG. 5) protrudes from the tip of the base metal 300. The tool main body 400 is fixed to a holder of the cutting machine (not shown), and the monocrystal diamond cutting tool 200 can cut an object to be cut to form the groove therein as described later.

[0054] As illustrated in FIG. 5, the monocrystal diamond cutting tool 200 has a rake surface 201, two first flanks 203 and 204 that are side surfaces each adjacent to the rake surface 201, a front flank 205 that is adjacent to the rake surface 201 and is opposed to a cutting surface of an object to be cut, a front cutting blade 210 formed at a boundary between the rake surface 201 and the front flank 205, and two first cutting blades 213 and 214 formed at a boundary between the rake surface 201 and the first flank 203 and between the rake surface 201 and the first flank 204. Shapes of the front flank 205 and the rake surface 201 are not limited, and the shapes may be a flat surface or a curved surface. The rake surface 201 has a predetermined rake angle of zero degrees or a rake angle slightly inclined in a positive direction so as to scoop cutting waste.

**[0055]** The first flank 203, 204 or the front flank 205 is formed by etching with a focused ion beam (FIB). The etching with the FIB enables machining of a complicated shape, and has an advantage that any crystal surface can be machined. Therefore, even a (111) plane of diamond that is the hardest crystal plane can be easily machined. In contrast, in a case of polishing with a grindstone, for example, the (111) plane of diamond cannot be polished.

[0056] It is preferred to set a width W1 of the front cutting blade 210 to 4  $\mu m$  or smaller because a fine groove portion suitable for the diffraction grating for the X-ray Talbot interferometer can be formed.

[0057] The monocrystal diamond cutting tool 200 (tool

main body 400) described above is mounted to the cutting machine, and can form the groove portion 20a as illustrated in FIG. 6. Here, the period and the depth of the groove portions formed through cutting depend on a resolution of the cutting machine, and hence it is preferred to use the superfine nano machine having a nanometer-level resolution as the cutting machine. As the superfine nano machine, FANUC ROBONANO  $\alpha$ -0iB manufactured by FANUC CORPORATION, for example, is commercially available. This superfine nano machine has a resolution of 1 nm in a linear axis by controlling a linear motor and a synchronous built-in servomotor so that five axes are directly driven simultaneously with high accuracy.

[0058] Using such a machine, as illustrated in FIG. 6, the monocrystal diamond cutting tool 200 performs a drag cutting process on a metal film 20x along one direction (an arrow direction in FIG. 6) leaving the ridges having a predetermined width W3 in the direction perpendicular to the one direction. Thus, the metal film 20x is cut to form the groove portions 20a, and the ridge-like X-ray absorbing portion 20b can be formed between the neighboring groove portions 20a. Note that, it is preferred that W1 =W3 be satisfied.

[0059] Here, the monocrystal diamond cutting tool 200 cuts the metal film 20x to form the groove portion 20a, and hence it is possible to perform appropriate machining, in which the roughness of side surfaces of the groove portion and the curvature radius of the cut corner portion formed by the side surface and the bottom surface of the groove portion are 0.1  $\mu$ m or smaller each. Compared with the conventional technique in which electroforming is performed in the fine groove (slit) formed using a resist resin to manufacture the diffraction grating, a diffraction grating with a high dimensional accuracy can be obtained.

[0060] In addition, the width of the groove portion 20a is the same as the width W1 of the front cutting blade 210, and a depth of the groove portion 20a is equal to or smaller than a vertical length L of the first flanks 203 and 204. In this case, in order to manufacture the second diffraction grating (amplitude diffraction grating) illustrated in FIG. 1, it is necessary to set the aspect ratio expressed by (depth of groove)/(width of groove) to a high value (for example, three or higher). Therefore, in order to manufacture the diffraction grating through one cutting operation, it is necessary to use an elongated monocrystal diamond cutting tool having an aspect ratio L/W1 of three or larger. However, when the aspect ratio L/W1 is three or larger, the cutting tool may be broken easily.

[0061] Therefore, in the present invention, the depth

of the groove portion 20a formed through one cutting operation is set to a value that is not extremely large (the aspect ratio is set to a value that is not extremely high), and the unit metal layers formed through individual cutting operations are laminated. Thus, the aspect ratio of the finally obtained X-ray absorbing portion (groove portion) is high, and break of the cutting tool is reduced.

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**[0062]** For instance, when the monocrystal diamond cutting tool having an aspect ratio L/W1 of one is used, the aspect ratio of the groove portion obtained through one cutting operation is one. However, when the cutting operation is performed three times as described below, the diffraction grating having the groove portion with an aspect ratio of three or higher can be obtained.

**[0063]** Next, with reference to FIGS. 7A to 7D and FIGS. 8E to 8I, an example of a method of manufacturing the diffraction grating 20 for the X-ray Talbot interferometer is described.

[0064] First, the intermediate layer 24 is formed on the silicon wafer substrate 22 as necessary, and then a metal film (referred to as "initial metal film") 2bx is formed on the intermediate layer 24 (FIG. 7A; initial metal film forming step). As the intermediate layer 24, an aluminum film is formed by ion plating or sputtering vapor deposition. When the intermediate layer 24 is formed of copper, electric copper plating is used. A thin chromium vapor deposition film or titanium vapor deposition film may be formed between the substrate 22 and the intermediate layer 24 for securing adhesiveness of the intermediate layer. As the initial metal film 2bx, a gold film is formed by electric plating. In order to secure adhesiveness of the initial metal film 2bx, it is possible to form a thin gold film on the surface of the intermediate layer 24 by vapor deposition, and then to perform electric plating of gold.

**[0065]** Next, with use of the above-mentioned monocrystal diamond cutting tool 200, the initial metal film 2bx is cut along one direction (direction perpendicular to the drawing sheet of FIGS. 7A to 7D) at predetermined intervals so that the plurality of groove portions 20a are formed, and a ridge-like unit metal layer 2b is formed between the neighboring groove portions 20a (FIG. 7B; cutting step).

**[0066]** Then, a resin 26ax is filled in the groove portion 20a (FIG. 7C; resin filling step). Here, the resin 26ax completely fills the groove portion 20a and covers the surface of the unit metal layer 2b.

**[0067]** As described above, it is preferred to use at least one or both of polyimide and a para-xylene polymer as the resin 26ax. When polyimide is used, it is preferred to fill polyimide in the groove portion 20a by the vacuum injection method because polyimide can flow into a deep portion of the groove portion 20a. In addition, when the para-xylene polymer is used, it is preferred to fill the para-xylene polymer in the groove portion 20a by the vacuum vapor deposition method because the para-xylene polymer can be deposited in the deep portion of the groove portion 20a.

**[0068]** Note that, the vacuum injection method is a method in which the substrate 22 having the groove portions 20a is disposed in a system maintained in vacuum, and low molecular weight polyimide is supplied to the system to flow into the groove portion 20a and then is polymerized by ultraviolet rays or heat to cause a crosslinking reaction.

[0069] In addition, when the para-xylene polymer is

filled by the vacuum vapor deposition, a dimer of paraxylene is heated to approximately 175°C to evaporate in the vacuum. After that, the vapor of the para-xylene dimer is heated to approximately 680°C into thermally decomposed substances. When the thermally decomposed substances of para-xylene reaches the surface of the groove portion 20a or the X-ray absorbing portion 20b, the thermally decomposed substances are reacted with each other to be a stable vapor deposition film.

[0070] Next, the surface of the resin 26ax is ground or ashed so that the upper surface of the unit metal layer 2b is exposed (FIG. 7D; metal layer exposing step). When the grinding is performed in the metal layer exposing step, a resin 26a (the ground or ashed resin is represented by reference symbol 26a to be distinguished from the resin 26ax) and the unit metal layer 2b become substantially flush. However, when the ashing is performed, a slight difference in height may be generated therebetween. In the grinding process, for example, a fine grindstone is moved to slide in the same direction as cutting of the groove 20a so that the resin pushed out from the upper part is ground off. In addition, the ashing can be performed by using a known ashing apparatus (technique) used in semiconductor technology, and for example, oxygen plasma ashing can be used.

[0071] Next, a second metal film 4bx is laminated on the upper surface of at least the unit metal layer 2b (FIG. 8A; metal film laminating step). Here, the second metal film 4bx is a film for forming the unit metal layer 4b, and is formed ideally only on the upper surface of the unit metal layer 2b. Therefore, when the second metal film 4bx is formed by the electric plating using the unit metal layer 2b as a cathode, the second metal film 4bx is electrically deposited on the unit metal layer 2b and its vicinity, but the second metal film 4bx is not formed on the surface of the resin 26, which is preferred. A composition of the second metal film 4bx may be the same as that of the unit metal layer 2b.

[0072] Next, with the use of the above-mentioned monocrystal diamond cutting tool 200, the second metal film 4bx is cut along the side wall (2s in FIG. 3) of the unit metal layer 2b so as to form the plurality of groove portions 20a and the ridge-like unit metal layer 4b between the neighboring groove portions 20a (FIG. 8B; second cutting step). Here, when the above-mentioned superfine nano machine is used as the cutting machine, the second metal film 4bx can be cut so as to match the side wall 2s of the unit metal layer 2b by nanometer level. In addition, the groove portion 20a in the second cutting step is formed on the resin 26 exposed in the metal layer exposing step (FIG. 7D).

**[0073]** Note that, when the second metal film 4bx is formed by the electric plating, the surface of the second metal film 4bx becomes slightly convex (not flat). Therefore, it is preferred to set the thickness of the second metal film 4bx larger than a thickness designed as the unit metal layer 4b, and after the second cutting step, or in the second metal layer exposing step (FIG. 8D) de-

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scribed later, grind the surface of the second metal film 4bx to be adjusted to the thickness of the unit metal layer 4b and form a flat surface of the unit metal layer 4b.

[0074] Next, similarly to the above-mentioned resin filling step (FIG. 7C), a resin 26bx is filled in the groove portion 20a formed in the second cutting step (FIG. 8C; second resin filling step). Then, similarly to the above-mentioned metal layer exposing step (FIG. 7D), the surface of the resin 26bx is ground or ashed so that the upper surface of the unit metal layer 4b is exposed (FIG. 8D; second metal layer exposing step).

[0075] In this way, through repetition of the initial metal film forming step (FIG. 7A) and the cutting step (FIG. 7B) one or more times, it is possible to manufacture the diffraction grating for an X-ray Talbot interferometer including the X-ray absorbing portion in which two or more unit metal layers are laminated. In addition, it is preferred to perform the metal film laminating step (FIG. 8A), the second cutting step (FIG. 8B), the second resin filling step (FIG. 8C), and the second metal layer exposing step (FIG. 8D), after performing the resin filling step (FIG. 7C) and the metal layer exposing step (FIG. 7D).

[0076] Note that, in the case of the diffraction grating 20 for an X-ray Talbot interferometer illustrated in FIG. 3, the metal film laminating step (FIG. 8A), the second cutting step (FIG. 8B), the second resin filling step (FIG. 8C), and the second metal layer exposing step (FIG. 8D) are repeated two times, and hence it is possible to manufacture the diffraction grating 20 for an X-ray Talbot interferometer having the X-ray absorbing portion 20b in which three unit metal layers 2b, 4b, and 6b are laminated, and the resin 26 interposed in the groove portion 20a between the neighboring X-ray absorbing portions (FIG. 8I).

[0077] Note that, when an alignment mark 22a is disposed in peripheral regions of the surface of the substrate 22, in which the groove portion 20a and the like are not formed (see FIG. 9), so that the cutting position of the metal film is adjusted with reference to the alignment mark 22a, after the substrate 22 is transferred in each process step (for example, the metal layer exposing step (FIG. 7D), the substrate 22 can be carried back to the cutting machine at a precise position, and hence the second metal film 4bx can be cut precisely along the side wall 2s of the unit metal layer 2b. It is preferred to dispose the alignment mark 22a at three or more different positions on the surface of the substrate 22.

**[0078]** It should be understood that the present invention is not limited to the embodiment described above, and that the present invention incorporates various modifications within the scope of the present invention.

### Claims

 A diffraction grating (20) for an X-ray Talbot interferometer (100), comprising: a plurality of ridge-like X-ray absorbing portions (20b) made of a metal and formed on a substrate (22) along one direction at predetermined intervals; and

a resin (26) interposed in a groove portion (20a) between neighboring X-ray absorbing portions of the plurality of ridge-like X-ray absorbing portions, wherein:

the plurality of ridge-like X-ray absorbing portions are each formed of at least two unit metal layers (2a, 4a, 6a) laminated perpendicularly to a surface of the substrate; the at least two unit metal layers are formed through cutting of a metal film; and roughness of a side wall (2s) of the groove portion of each of the at least two unit metal layers has an arithmetical mean height Ra of 0.1  $\mu m$  or smaller, as defined in JIS B0601.

- 2. A diffraction grating for an X-ray Talbot interferometer according to claim 1, wherein each of the plurality of ridge-like X-ray absorbing portions (20b) contains gold as a main component and is formed of one of crystals each having an average grain diameter of 0.1 μm or smaller and amorphous gold.
- **3.** A method of manufacturing a diffraction grating (20) for an X-ray Talbot interferometer (100), the method comprising:

forming an initial metal film (2bx) on a substrate (22):

cutting the initial metal film along one direction at predetermined intervals so as to form a plurality of first groove portions (20a) and to form a ridge-like first unit metal layer (2b) between neighboring groove portions of the plurality of first groove portions;

filling a resin (26ax) in each of the plurality of first groove portions;

exposing an upper surface of the ridge-like first unit metal layer through one of grinding and ashing of a surface of the resin;

laminating a metal film (4bx) on at least the upper surface of the ridge-like first unit metal layer; and cutting the metal film along a side wall (2s) of the first ridge-like unit metal layer so as to form a plurality of second groove portions (20a) and to form a ridge-like second unit metal layer (4b) between neighboring groove portions of the plurality of second groove portions,

wherein the laminating the metal film and the cutting the metal film are repeated at least one time.

4. A method of manufacturing a diffraction grating for

an X-ray Talbot interferometer according to claim 3, wherein the cutting the metal film comprises adjusting a cutting position of the metal film with reference to an alignment mark (22a) disposed on a surface of the substrate.

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**5.** A method of manufacturing a diffraction grating for an X-ray Talbot interferometer according to claim 3 or claim 4, further comprising forming an intermediate layer (24) between the substrate (22) and the initial metal film (2bx), the intermediate layer having a smaller hardness than the substrate and containing an element having a smaller effective atomic number than an element contained in the initial metal film.

6. A method of manufacturing a diffraction grating for an X-ray Talbot interferometer according to any one of claims 3 to 5, wherein the resin comprises at least one of polyimide and a para-xylene polymer.

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7. A method of manufacturing a diffraction grating for an X-ray Talbot interferometer according to any one of claims 3 to 6, wherein groove portions are filled at least once with polyimide as the resin by a vacuum

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injection method.

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8. A method of manufacturing a diffraction grating for an X-ray Talbot interferometer according to any one of claims 3 to 7, wherein groove portions are filled at least once with a para-xylene polymer as the resin by a vacuum vapor deposition method.

9. A method of manufacturing a diffraction grating for an X-ray Talbot interferometer according to any one of claims 3 to 8, wherein the cutting the initial metal film and the cutting the metal film comprises cutting using a cutting tool (400) having a cutting blade (200) made of monocrystal diamond.

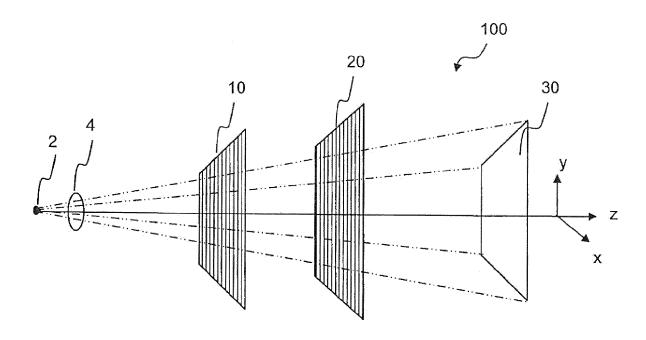
10. An X-ray Talbot interferometer (100), comprising a diffraction grating for an X-ray Talbot interferometer according to any of claims 1 to 3.

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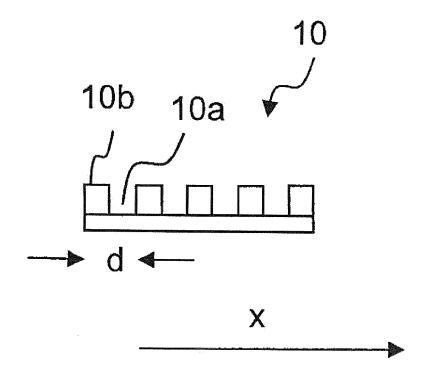
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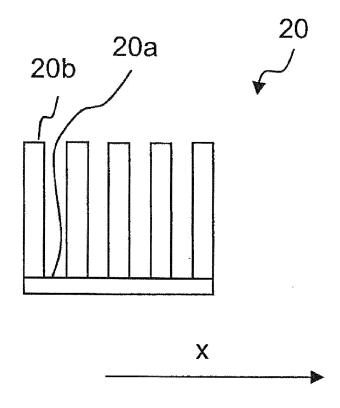
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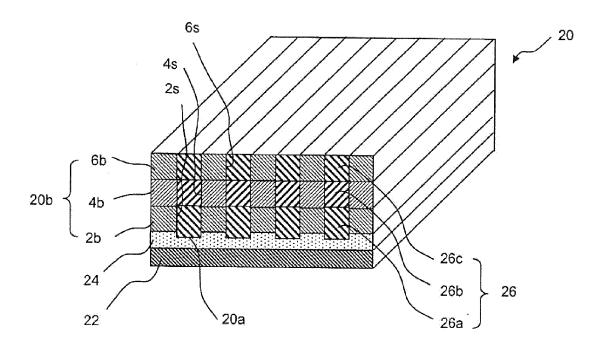
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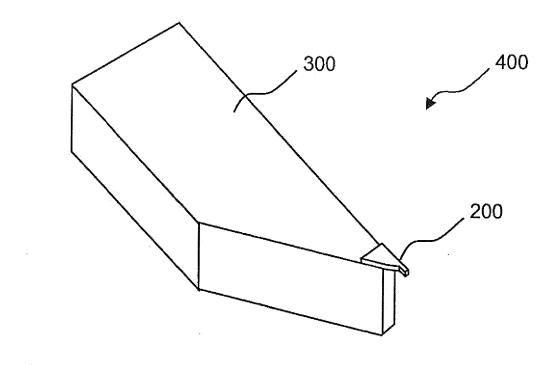
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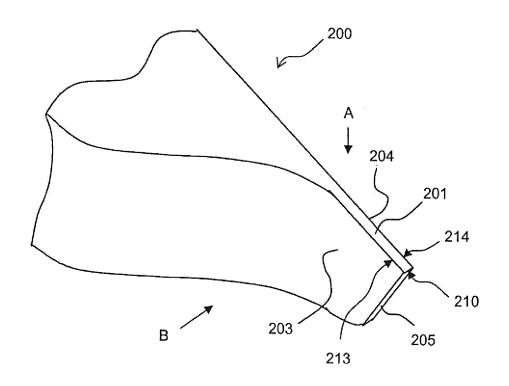
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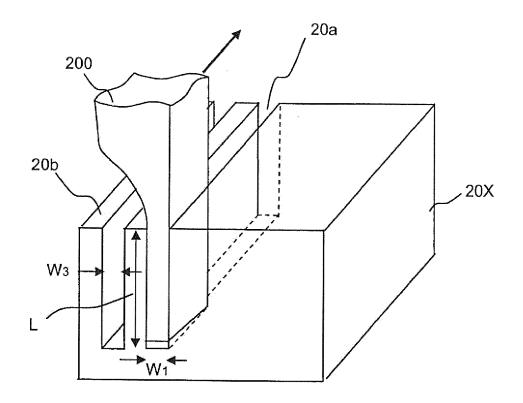
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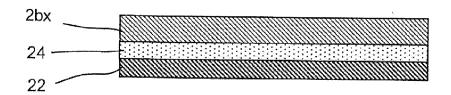


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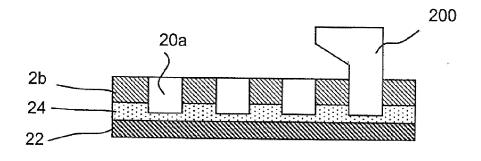


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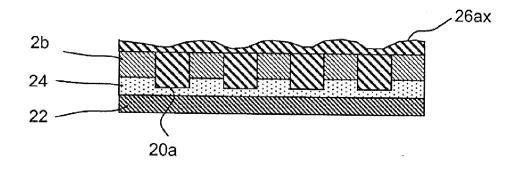
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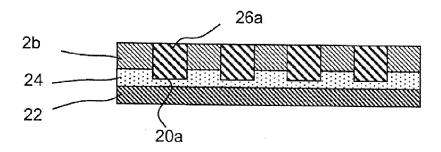
F I G. 7 B



F I G , 7 C

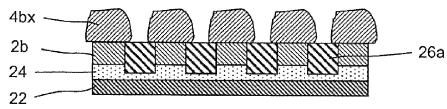


F I G. 7 D

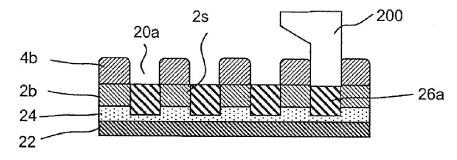


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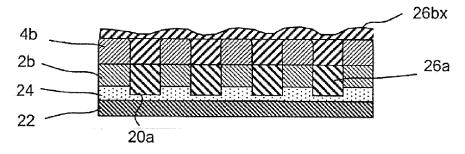




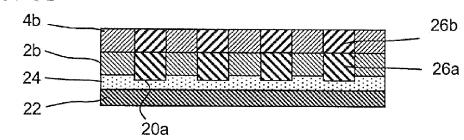
F I G. 8 B



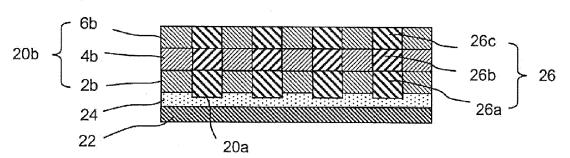
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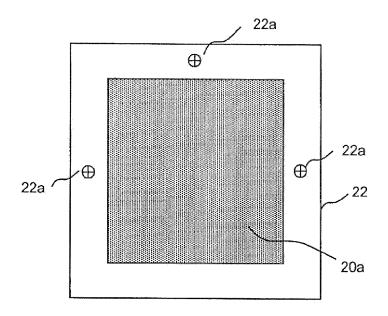
F I G. 8 D



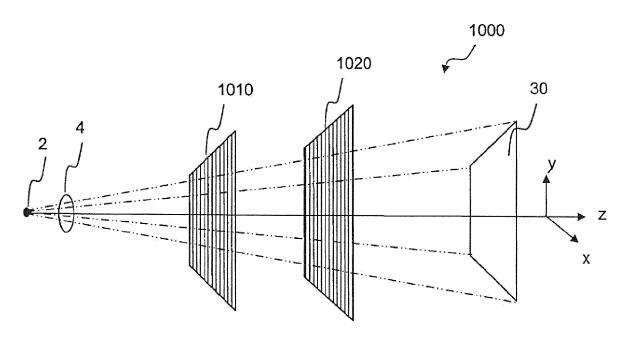
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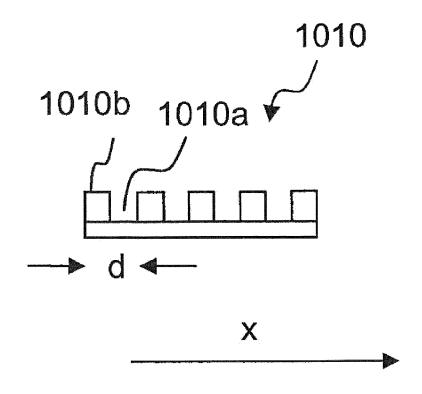
F I G. 9



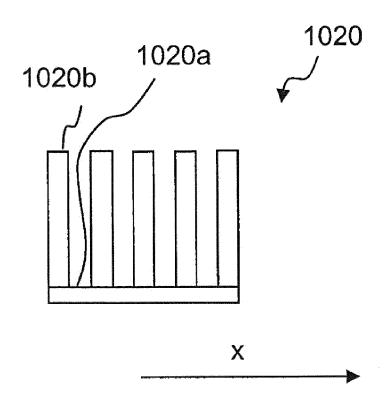
F I G . 1 0



F I G. 1 1 A



F I G. 11B



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#### REFERENCES CITED IN THE DESCRIPTION

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