(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) **EP 2 168 130 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention
of the grant of the patent:
06.06.2012 Bulletin 2012/23

(21) Application number: **08773243.4**

(22) Date of filing: **19.06.2008**

(51) Int Cl.:
G21K 1/06 *(2006.01)*

(86) International application number:
PCT/CZ2008/000072

(87) International publication number:
WO 2009/012732 (29.01.2009 Gazette 2009/05)

(54) OPTICAL ELEMENT FOR X-RAY MICROSCOPY

OPTISCHES ELEMENT FÜR RÖNTGENMIKROSKOPIE

ELÉMENT OPTIQUE POUR UNE MICROSCOPIE PAR RAYONS X

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MT NL NO PL PT RO SE SI SK TR

(30) Priority: **20.07.2007 CZ 20070494**

(43) Date of publication of application:
31.03.2010 Bulletin 2010/13

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(56) References cited:
JP-A- 6 232 015
US-A- 5 210 779
US-A1- 2003 147 161

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Printed by Jouve, 75001 PARIS (FR)

Description

Technology Field

[0001] Presented solution deals with an X-ray optical element suitable in particular for X-ray microscopy and other X-ray-based display systems.

Existing Conditions of Technology

[0002] X-ray radiation means an electromagnetic radiation with wavelengths shorter than 10 nm. Until now, instead of optical lenses used in visible part of light spectrum, X-ray microscopy uses as display elements Fresnel zone plates, biconcave refractive X lenses, polycapillar optics or Göbel mirrors.

[0003] Fresnel zone plates are Fresnel lenses produced by means of electron lithography. Using lithographic methods - but other manufacturing procedures are possible as well - ringlets, or parts of concentric paraboloids, are created in such a way to ensure that radiation emitted from a point on the ringlets' central axis (optical axis), having passed through the ringlets, is focused back again into a point lying on this axis. Thickness of these ringlets or their parts is in orders of tens of nm and their height is roughly 1000 nm. These plates are used as radiation condensers for scanning and display microscopes; in display microscopes they are used also as lenses.

[0004] Another possible way to produce Fresnel zone plates is to create a set of little plates with separate segments containing parallel strips with identical spacing between each other. Diameters of such optical elements are several centimeters as a maximum.

[0005] Biconcave refractive X lenses take the advantage of the fact that the refractive index of certain materials for X-ray radiation is only slightly higher than one and a set of identical cavities, located one after another, creates a condensing lens with long focal distance. In practice, two crossed systems of biconcave lenses are often used, each focusing the radiation in one direction only. The result is a focusation into a point, which, owing to little differences between the refractive index of the matter and the air, is located in relatively long distance from the lenses - in order of meters.

[0006] Polycapillar optics is created by a set of curved capillaries, which lead the X-ray radiation and focus it into a point.

[0007] Göbel mirrors are plates formed by alternated thin layers of two metals, such as wolfram and silicon, with a shape of part of the surface of elliptic spheroid. These mirrors focus monochromatic X-ray radiation into a point.

[0008] All optical elements described above feature big ratio of focal distance to their diameter, it means relatively long focal distances and small diameters, and their production is costly. They are used in X-ray microscopy for normal as well as so-called scanning microscopes. In scanning microscopes, the beam is focused by the optics into a point on a specimen and a detector senses intensity of X-ray radiation, which has passed through. By measuring all points - which is carried out by shifting the specimen in two mutually orthogonal directions-specimen's final image in given wavelength is obtained. Information about intensity of the radiation from the X-ray radiation detectors or digital sensors, such as CCD cameras, which may be used instead of detectors, is led into a computer where the final image is processed.

[0009] All examples described above result in relatively high absorption rate of X-ray radiation and all systems feature also relatively long focal distances, i.e. distances between the display and object, and they can be used for longer wavelengths, around 10 nm. Also, these components impose high demands on production equipment and therefore they are rather expensive.

[0010] There exist also the following documents defining general state of the art, but they do not solve the problem which is subject-matter of this patent application.

[0011] The device described in JP 06 232015 A is used for marking the plane orientation of a single crystal ingot, not for focusing X-rays.

[0012] US 5 210 779 A describes a hard X-rays focusing method using dislocation free silicon - germanium crystal, wherein the proportion of germanium and silicon varies over the length of the crystal and change the lattice parameter - we use the stress and shape for focusing X rays.

[0013] US 2003/147161 A1 relates to a method using stress on pheripheral - side surface of the mirror to provide concave or convex deviation on reflective surface of mirror, in order to form errors arising on surface.

Summary of the Invention

[0014] All disadvantages mentioned above are improved by an optical set-up exploiting a monocrystal, which displays X-ray radiation with wavelength λ according to the presented solution. This set-up consists of at least one monocrystal with atomic planes in parallel with optical axis, it means with a line connecting the point to be displayed with the center of its image. Mutual distance of the atomic planes in resisting state without the force is d_0. Cross section of the monocrystal is variable. With respect to the optical axis the farther and/or closer side of monocristal, which is orthogonal to this optical axis, is equipped with a device to create a pull or push force and to maintain a pull or push strength in direction orthogonal to atomic planes of such monocrystal. Size of cross section S of the monocrystal under action of pull or push force in distance R from optical axis is directly proportional to pre-selected cross section So and push or pull force F is directly proportional to modulus of elasticity E of given monocrystal in direction of force F in action. Size of the monocrystal's cross section is given by the following formula

$$S = \frac{S_0 R\left(\left(R_0^2 + s^2\right)^{0.5} - 1\right)}{R_0\left(\left(R^2 + s^2\right)^{0.5} - 1\right)}$$

and the force F is defined by the equation

$$F = \pm S_0 E\left(\frac{n\lambda}{2R_0 d_0}\left(R_0^2 + s^2\right)^{0.5} - 1\right)$$

where s, with respect to the monocrystal's longitudinal axis, is an object and also an image distance and So is a pre-selected cross section of monocrystal upon requirements of the application in distance R_0 from the optical axis and n is a natural number. In the equation, the + sign applies for pull force, the - sign applies for push force. Further, as already mentioned above, λ is the X-ray radiation wavelength and So is a pre-selected cross section in distance R_0.

[0015] In another possible embodiment, the optical set-up is formed by minimum two identical monocrystals arranged around the optical axis. All such monocrystals feature equal object and display distance s, while each monocrystal is equipped with a device applying push or pull force F within the minimum distance R_0 from the optical axis and the maximum distance R_m from the optical axis. Their cross section is calculated by means of the above-mentioned equation. In other words, it is possible to arrange the monocrystals around the optical axis provided their object and display distances for the same wavelength are equal.

[0016] If cross section of the monocrystal's farther side the from the optical axis is bigger than cross section of the monocrystal's closer side to this optical axis, at least one of these sides is equipped with a device creating a pull force between the both sides. On the contrary, if cross section of the monocrystal's farther side the from the optical axis is smaller than cross section of the monocrystal's closer side to this optical axis, at least one of these sides is equipped with a device creating a push force between the both sides.

[0017] Advantage of this solution is relatively simple production of its components, which is based on Hooke's law without needing to know the exact position of individual atomic planes. Production of current Fresnel structures involves electron lithography for individual ringlets, while this method requires only precise machining of the outer shape, for instance cutting by water jet or laser, and applying a force, which can be controlled. Machining of the outer shape results in better physical effect, it means lower absorption and larger active area, with lower costs because lithography is significantly more expensive than cutting. Another advantage is that it works for also for lower order wavelengths, i.e. below 1 nm.

Overview of Figures in Drawings

[0018] Example of a displaying optical set-up according to the presented solution is shown in attached drawings. Fig. 1 and 2 show examples of creating two different monocrystals. Fig. 3 shows example of basic displaying set-up. Fig. 4 shows utilization of the set-up for rotational X-ray scanning microscope. Fig. 5 shows a method of creating an optical element from multiple monocrystals and fig. 6 shows its exploitation within a set-up. Fig. 7 shows a chart of dependence of sections ratio S/S_0 on distance from optical axis used for determination of pull force on atomic planes.

Detailed Description of the Preferred Embodiments

[0019] Fig. 1 and 2 show two examples how monocrystal 1 can be created, with designated atomic planes 2. Fig. 1 schematically shows possible shape of monocrystal 1, an asymmetric shape, designed for shifting the atomic planes by push force. Monocrystal's 1 shape in fig. 2 is designed for shifting the atomic planes by pull force. In all cases, the atomic planes 2 where the beam reflects or refracts are horizontal. The first example shows a monocrystal 1 where optical axis, i.e. a line connecting a point to be displayed with center of its image, is located under the monocrystal's 1 bigger cross section and the arrow indicates a push force applied by push device. On the contrary, fig. 2 shows situation when the monocrystal's 1 imaginary optical axis is located under the monocrystal's 1 smaller cross section and the arrow indicates a pull force applied by pull device. Push and pull devices may be implemented in various ways, such as a fixed point and piezo crystal, screw mechanism and tension gauge, etc., and they can be located on one or both opposite sides of monocrystal 1.

[0020] Fig. 3 shows example of an optical set-up displaying X-ray radiation with wavelength λ. This set-up consists of monocrystal 1 with atomic planes 2, which are arranged in parallel with optical axis 3. Mutual distance of atomic planes 2 in resting state is do and the monocrystal's 1 cross section S is variable. On the farther side of monocrystal 1 from the optical axis 3, orthogonal to this optical axis 3, the cross section is smaller than on the side closer to the optical axis 3, and therefore it is equipped with a push device, which is not shown in the figure. Purpose of this push device is to maintain a push force F in direction orthogonal to atomic planes 2 of this monocrystal 1.

[0021] The figure schematically shows function of symmetrically processed monocrystal 1, exposed to push force, which focuses X-ray radiation with wavelength λ from distance s to distance s. Both these distances must be equal. Monocrystal 1 is located between distances R_0 and R_m from optical axis 3 where R_0 is the minimum distance and R_m is the maximum distance from optical axis 3. Based on given equation, cross section S, which is not shown in the figure in order to keep it clear but which can be seen in fig. 1 and 2, is a function of distance R.

[0022] Cross section S of monocrystal 1 in distance R from optical axis 3 is directly proportional to a pre-selected pull or push force F and indirectly proportional to modulus of elasticity E of given monocrystal 1 in direction of force F in action and depends on distance R and is given by the following formula.

$$S = \frac{S_0 R\left(\left(R_0^2 + s^2\right)^{0.5} - 1\right)}{R_0\left(\left(R^2 + s^2\right)^{0.5} - 1\right)} \qquad (1)$$

where

$$F = \pm S_0 E\left(\frac{n\lambda}{2R_0 d_0}\left(R_0^2 + s^2\right)^{0.5} - 1\right) \qquad (2)$$

where s, with respect to the monocrystal's 1 longitudinal axis, is an object and also a display distance, n is natural number. The plus sign applies for pull force, the minus sign applies for push force applied onto monocrystal 1, while value of S will always be positive. A is the X-ray radiation wavelength and S_0 is a pre-selected cross section of monocrystal 1 upon requirements of the application in distance R_0 from the optical axis 3.

[0023] Described device is built as follows. Based on used source of X-ray radiation the wavelength A is selected for microscopy as a wavelength, at which the source radiates with maximum intensity, or according to other requirements for application and parameters of monocrystal 1 the object and display distance s and cross section S_0 are selected, for which the force F is calculated for minimum distance R_0 of the monocrystal 1 from optical axis 3. Subsequently, the dependence of cross section S on distance R is additionally calculated according to the given equation based on calculated force F and modulus of elasticity E. A plate with shape complying with the equation is cut from monocrystal 1. Rays emitted from a point at the object impact the monocrystal 1, reflect from individual atomic planes 2 and are projected in a display plane as shown in fig. 3.

[0024] In case of rotating X-ray scanning microscope with a source 4 of X-ray radiation, single monocrystal 1 may be used, embodied as shown in fig. 4, where the specimen 5 rotates around axis orthogonal to it, fixed in space, passing through the center of specimen 5. Such specimen 5 is after each revolution gradually shifted in direction of the arrow, thus allowing its scanning from one end to the other. Intensities of radiation measured by radiation detector 6 are processed by computer into the resulting image similarly as in case of tomography. Simultaneously, this monocrystal 1 acts a monochromator and a separate monochromator may thus be excluded.

[0025] As shown in fig. 6, optical set-up may consist of multiple identical monocrystals 1, which are arranged around the optical axis 3 as shown in fig. 5. All these monocrystals 1 have equal object and display distance, while each monocrystal 1 is equipped with a device applying on each monocrystal 1 pull force F between the minimum distance R_0 of the bottom side of monocrystals 1 from optical axis 3 and between maximum distance R_m from optical axis 3. Their cross section is again calculated according to the given equation. Shape of monocrystals 1 may vary provided their pre-selected cross sections are maintained. In this case, as an example, an optical element consisting of 28 monocrystals 1 is shown, where atomic planes 2 act directly as a set-up of concentric polygon ringlets. By selecting suitable shape and applying suitable force in compliance with the equation it is possible to achieve a situation when among the atomic planes 2 are such distances, which cause the same constructive interference as Fresnel lens for selected wavelength, or possibly also for wavelength, which is half of such wavelength, its third, etc., and act simultaneously as a monochromator, which selects only these wavelengths from the whole spectrum used. The same effect may be achieved by using suitable gradient of admixtures in crystal, such as hydrogen in gadolinium, which affects interatomic distance. Implementation of detectors is the same as in other cases.

[0026] Fig. 7 shows chart with dependencies of sections ratios S/S_0 of monocrystal 1 on distance from optical axis 3 used for determination of pull force on atomic planes 2 for the following values: $\lambda = 0.1$ nm, do = 0.4 nm, $R_0 = 0.01$ m, s = 0.5 m and n = 1, in the extent up to $R_{max} = 0{,}07$ m.

[0027] Described X-ray optical system exploits properties of spatial grid for focusing monochromatic X-ray radiation from a point source 4 into a point, fig. 6, or possibly a line segment, fig. 4, which may be achieved by altering the distance between atomic planes 2. Such shift of atomic planes 2 in monocrystal 1 is created by applying a tension, i.e. pull or push force F, orthogonal to atomic planes 2, and by suitable selection of cross section S in given height above optical axis 3, which is calculated using the above-mentioned equation.

Industrial Applicability

[0028] Described element may be used in particular in X-ray microscopy for focusing the X-ray radiation into a line segment. By arranging multiple pieces, an X-ray monochromatic lens for single wavelength may be created, which may serve as a condenser and a monochromator at the same time.

Claims

1. Optical set-up arrangeable for displaying X-ray radiation of wavelength λ such that the object distance s of a point to be displayed equals its image distance s, the point to be displayed with the center of its image defining an optical axis,

   the optical set-up comprising

   at least one monocrystal (1) with variable cross section S and with atomic planes (2), the monocrystal being arranged such that said atomic planes are in parallel with optical axis (3), and

   a device for creating and maintaining pull or push force F in direction orthogonal to said atomic planes (2) arranged at that side of the monocrystal (1) that is farther from the optical axis and/or at that side of the monocrystal (1) that is closer to the optical axis (3), where under the action of the pull or push force, the size of the cross section S of the monocrystal (1) at a distance R from the optical axis (3) is directly proportional to the pull or push force F and to the cross section S_θ at the distance R_θ from the optical axis (3), and is indirectly proportional to the modulus ef elasticity E of the monocrystal (1) in the direction of the force F,

   and wherein the variable cross section S of the least one monocrystal is given by the formula

$$S = \frac{S_0 R\left(\left(R_0^2 + s^2\right)^{0.5} - 1\right)}{R_0\left(\left(R^2 + s^2\right)^{0.5} - 1\right)},$$

   and the device for creating and maintaining the pull or push force is arranged to apply a force F given by the formula

$$F = \pm S_0 E\left(\frac{n\lambda}{2R_0 d_0} \cdot \left(R_0^2 + s^2\right)^{0.5} - 1\right),$$

   Where n is natural number, do is the mutual distance of said atomic planes in the resting state, and the plus sign applies for a pull force and the minus sign applies for a push force.

2. Optical set-up **according to claim 1** comprising a minimum two monocrystals (1), wherein all of these monocrystals

(1) are arranged around the optical axis (3) and wherein each monocrystal (1) is equipped with at least one device for applying and maintaining pull or push force F

3. Optical set-up according to claims 1 or 2 **characterized by the fact that** the cross section of the side of the monocrystal (1) that is farther from the optical axis (3) is bigger than the cross section of that side of the monocrystal (1) that is closer to this optical axis (3), and by the fact that at least one of these sides is equipped with a device arranged for creating a pull force between the both sides.

4. Optical set-up according to claims 1 or 2 **characterized by the fact that** the cross section of the side of the monocrystal (1) that is farther from the optical axis (3) is smaller than the cross section of that side of the monocrystal (1) that is closer to this optical axis (3), and by the fact that at least one of these sides is equipped with a device arranged for creating a push force between the both sides.

Patentansprüche

1. Optisches System geordnet für die Darstellung der Röntgenstrahlen der Wellenlänge λ so, dass die Gegenstandsweite (s) des dargestellten Punktes seiner Bildweite (s) gleich ist
dieses optische System enthält
mindestens einen Monokristall (1) mit veränderlichem Querschnitt (S) und mit atomaren Ebenen (2), wo dieser Monokristall so aufgebaut ist, dass diese atomaren Ebenen parallel zu der optischen Achse (3) sind,
und
die Anlage für Ausübung und Erhaltung der Zug- oder Druckkraft (F) in der senkrechten Richtung auf die atomaren Ebenen (2), die an der Seite des Monokristalls (1) platziert sind, die weiter von der optischen Achse ist und/oder an der Seite des Monokristalls (1), die näher zu der optischen Achse (3) ist, wo die Querschnittgröße (S) des Monokristalls (1) in der Entfernung (R) von der optischen Achse (3) bei der Wirkung der Zug- oder Druckkraft direkt proportional zu der Zug- oder Druckkraft und zu dem Querschnitt (S_0) in der Entfernung (R_0) von der optischen Achse (3) und indirekt proportional zu dem Elastizitätsmodul € des Monokristalls (1) in der Richtung der wirkenden Kraft (F) ist und wo der veränderliche Querschnitt (S) mindestens eines Monokristalls durch die Beziehung bestimmt ist,

$$S = \frac{S_0 R\left(\left(R_0^2 + s^2\right)^{0.5} - 1\right)}{R_0\left(\left(R^2 + s^2\right)^{0.5} - 1\right)}$$

und die Anlage für Ausübung und Erhaltung der Zug- oder Druckkraft ist für die Herleitung der Kraft (F) aufgebaut, die durch die Beziehung bestimmt ist,

$$F = \pm S_0 E\left(\frac{n\lambda}{2R_0 d_0}\left(R^2 + s^2\right)^{0.5} - 1\right)$$

wo n eine natürliche Zahl ist, d_0 die gegenseitige Entfernung der atomaren Ebenen im Ruhezustand ist, und das Vorzeichen plus die Zugkraft und das Vorzeichen minus die Druckkraft darstellt.

2. Optisches System entsprechend dem Anspruch 1 bestehend aus mindestens zwei Monokristallen, wo alle diese Monokristalle (1) um die optische Achse (3) angeordnet sind und wo jeder Monokristall (1) mit wenigstens einer Anlage für Ausübung und Erhaltung der Zug- oder Druckkraft (F) versehen ist

3. Optisches System entsprechend dem Anspruch 1 oder 2 wird **dadurch gekennzeichnet , dass** der Querschnitt der weither von der optischen Achse (3) entfernten Seite des Monokristalls (1) größer ist, als der Querschnitt der Monokristall-Seite, die näher der optischen Achse gelegen ist, und mindestens eine dieser Seiten ist mit der Vorrichtung für die Bildung der Zugkraft zwischen den beiden Seiten versehen.

4. Optisches System entsprechend dem Anspruch 1 oder 2 wird **dadurch gekennzeichnet, dass** der Querschnitt der

weither von der optischen Achse (3) entfernten Seite des Monokristalls (1) kleiner ist, als der Querschnitt der Monokristall-Seite, die näher der optischen Achse gelegen ist, und mindestens eine dieser Seiten ist mit der Vorrichtung für die Bildung der Druckkraft zwischen den beiden Seiten versehen.

Revendications

1. Le dispositif optique agencé pour afficher des rayons X de longueur d'onde λ de sorte que la distance d'objet (s) d'un point à afficher est égale à sa distance d'image (s), le point à afficher définissant l'axe optique par le centre de son image,
le dispositif optique agencé comprenant au moins un monocristal (1) avec la section transversale variable (S) et avec des plans atomiques (2) où le monocristal est agencé de telle sorte que ces plans atomiques sont parallèles avec l'axe optique (S),
et
un dispositif pour déclencher et maintenir la traction ou poussée de force (F) dans la direction orthogonale à ces plans atomiques (2) placé sur le côté de monocristal (1) plus éloigné de l'axe optique et / ou sur le côté de monocristal (1) plus proche de l'axe optique (3), où sous l'action de la force de traction ou de poussée, la taille de la section transversale (S) du monocristal (1) à la distance (R) de l'axe optique (3) est directement proportionnelle à la force de traction ou de poussée et à la section transversale (So) à la distance (Ro) de l'axe optique (3) et est indirectement proportionnelle au module d'élasticité (E) du mocristal (1) dans le sens de la force (F),
et où la section transversale variable (S) d'au moins un monocristal est donnée par la formule

$$S = \frac{S_0 R\left(\left(R_0^2 + s^2\right)^{0.5} - 1\right)}{R_0\left(\left(R^2 + s^2\right)^{0.5} - 1\right)}$$

et le dispositif de déclanchement et de maintien de la force de traction ou de poussée est agencé pour appliquer une force F selon la formule

$$F = \pm S_0 E\left(\frac{n\lambda}{2R_0 d_0}\left(R^2 + s^2\right)^{0.5} - 1\right)$$

où n est un nombre naturel, d_0 est la distance entre des plans atomiques à l'état de repos, le signe plus représente la force de traction et le signe moins représente la force de poussée.

2. Le dispositif optique selon la revendication 1, comprenant un minimum de deux monocristaux (1) où les monocristaux (1) sont disposés autour de l'axe optique (3) et où chaque monocristal (1) est équipé d'au moins un dispositif de déclanchement et de maintien de la force de traction ou de poussée (F).

3. Le dispositif optique selon les revendications 1 ou 2, **caractérisé par le fait que** la section transversale du côté de monocristal (1) plus éloigné de l'axe optique (3) est plus grande que la section transversale du côté de monocristal (1) plus proche de cet axe optique (3), et au moins un de ces côtés est muni d'un dispositif de déclanchement de la force de traction entre les deux côtés.

4. Le dispositif optique selon les revendications 1 ou 2, **caractérisé par le fait que** la section transversale du côté de monocristal (1) plus éloigné de l'axe optique (3) est plus petite que la section transversale du côté de monocristal (1) plus proche de l'axe optique (3), et au moins un de ces côtés est muni du dispositif de déclanchement et de maintien d'une force de poussée entre les deux côtés.

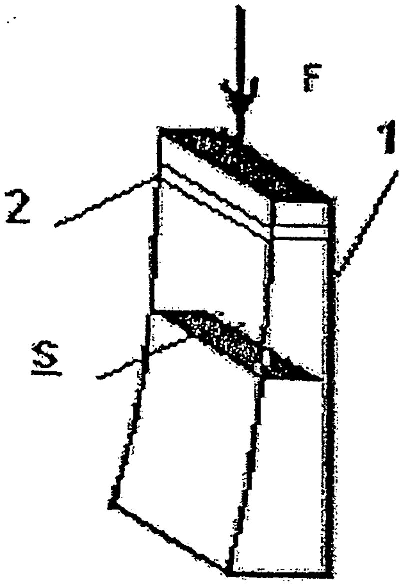

FIG.1

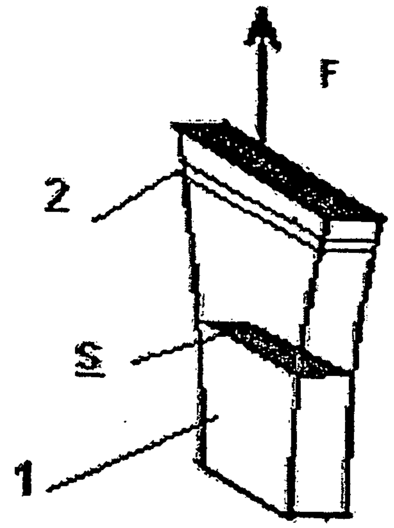

FIG.2

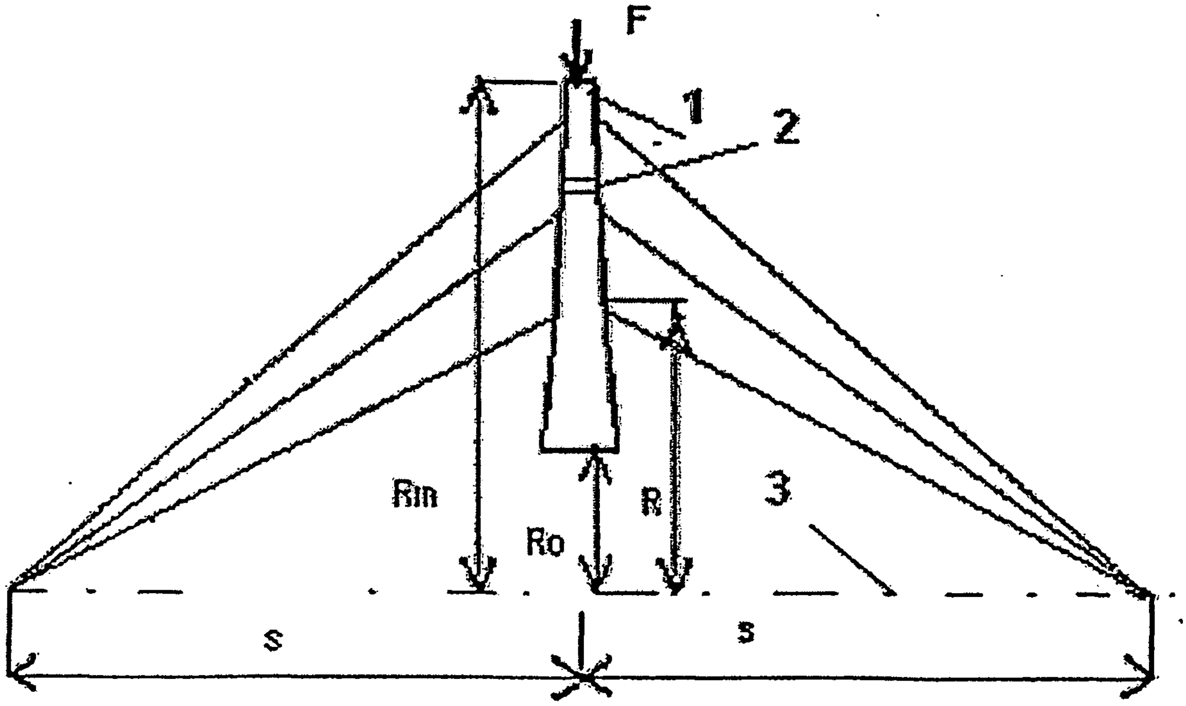

FIG.3

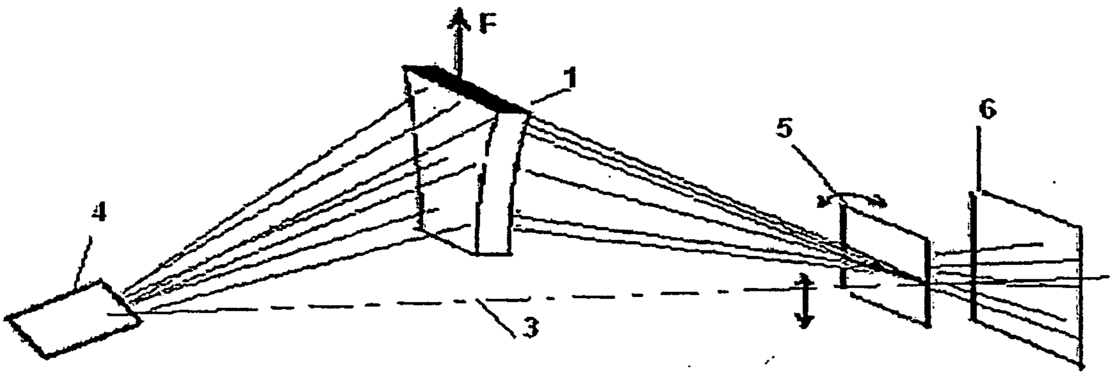

FIG. 4

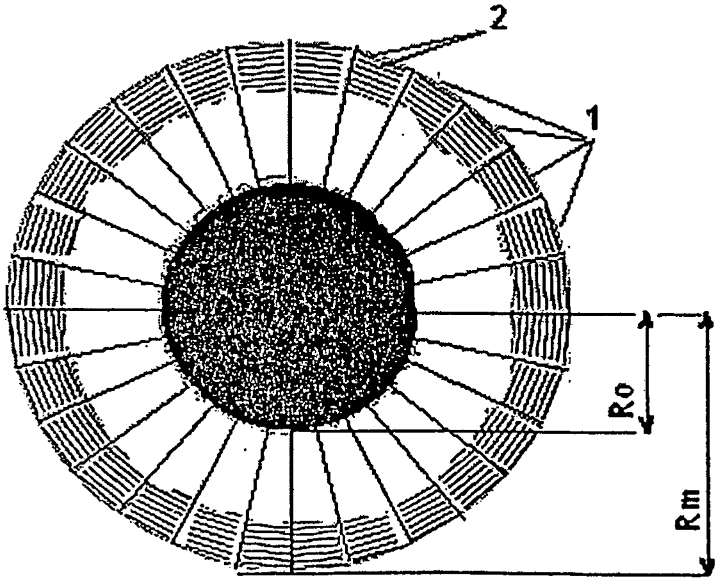

FIG. 5

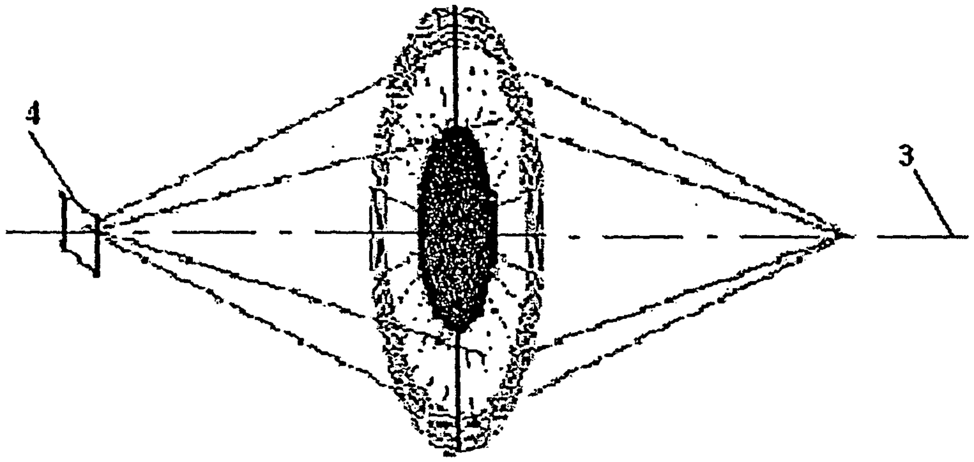

FIG. 6

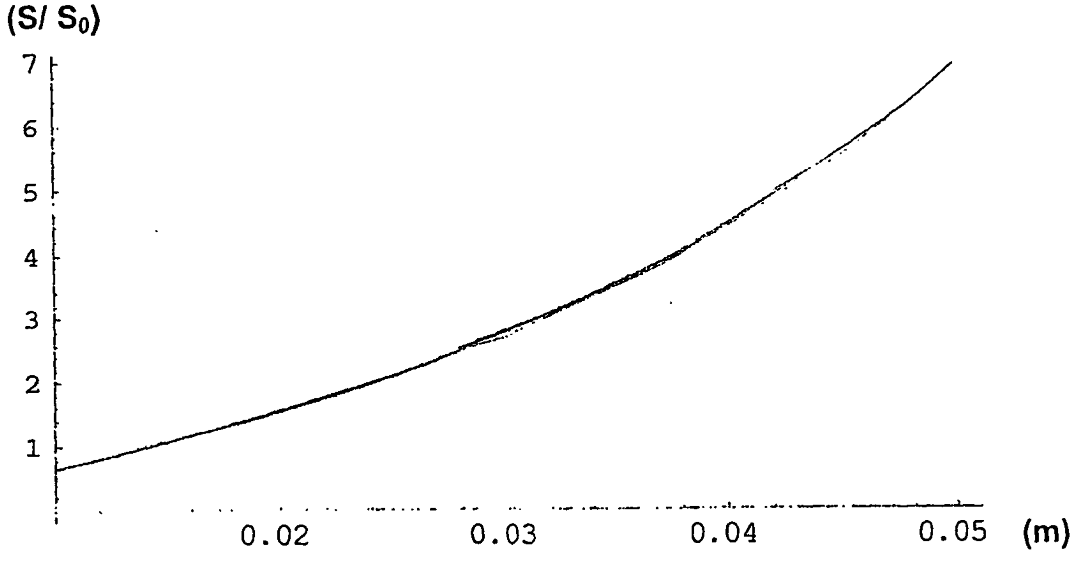

FIG. 7

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- JP 6232015 A **[0011]**
- US 5210779 A **[0012]**
- US 2003147161 A1 **[0013]**