May 1, 1962 R. HOSEMANN ET AL 3,032,656

X-RAY REFRACTING OPTICAL ELEMENT

Filed Aug. 15, 1957

Fig. 1

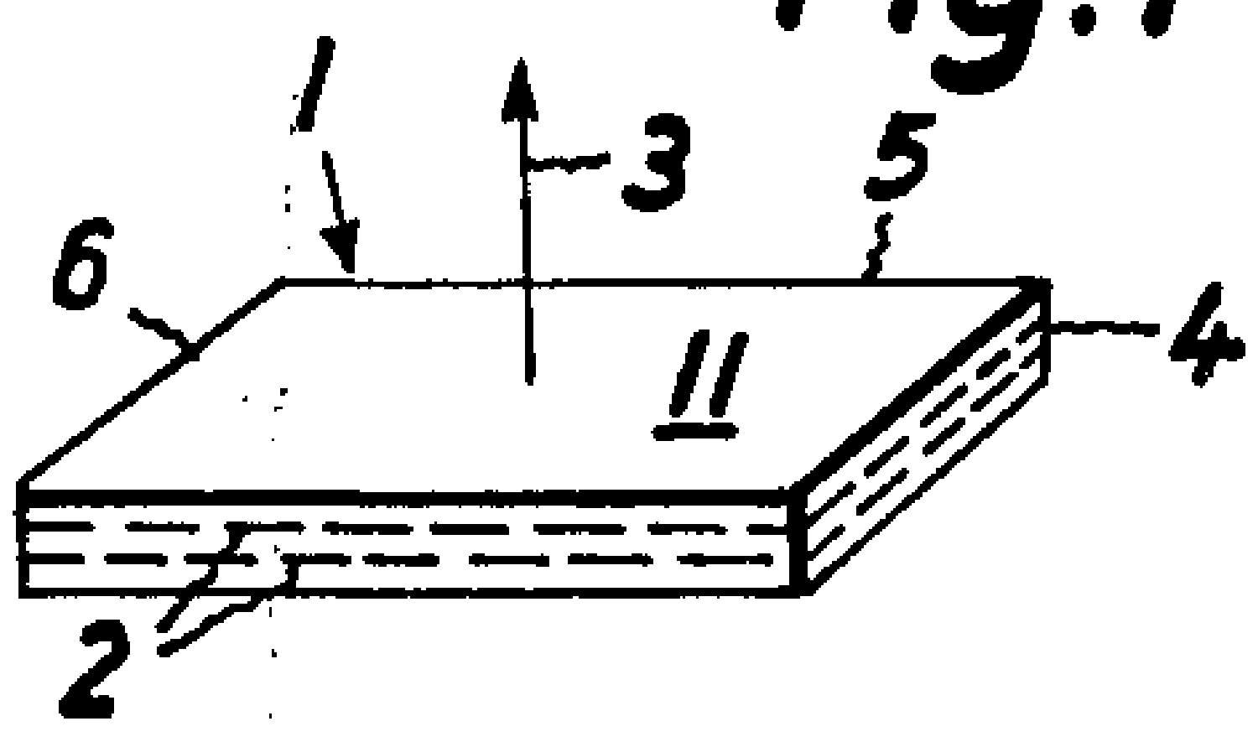


Fig. 2

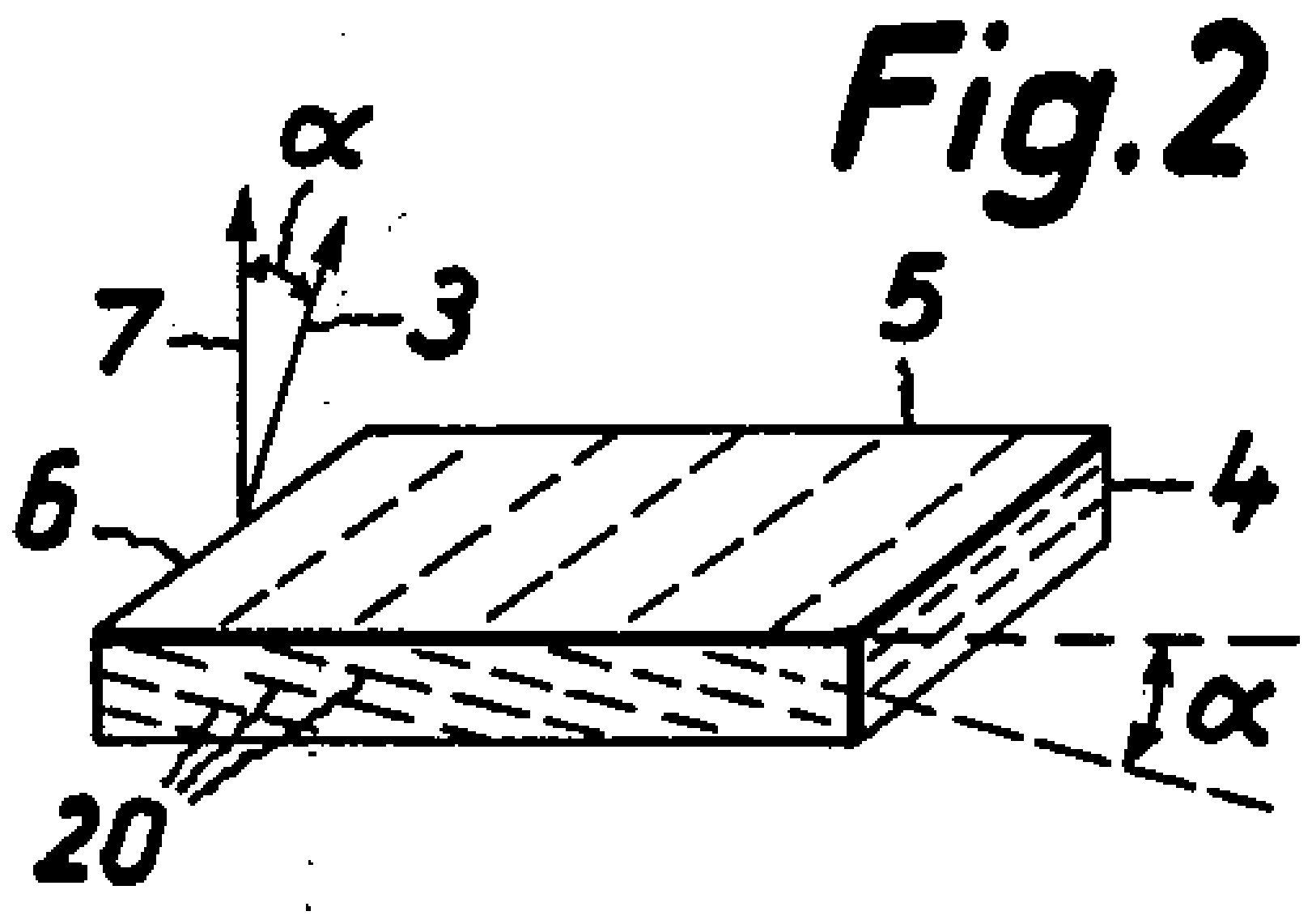


Fig. 3

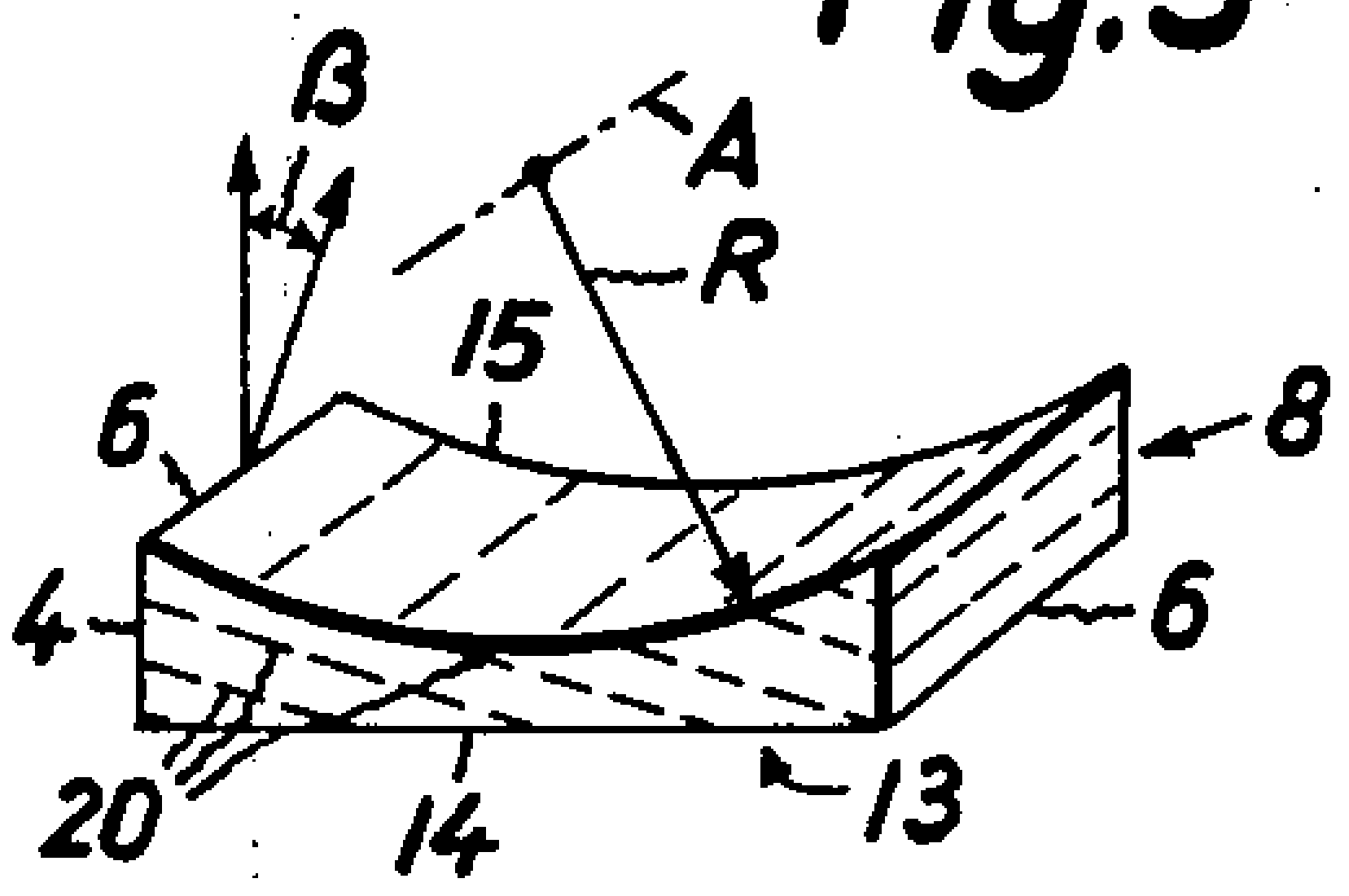


Fig. 4

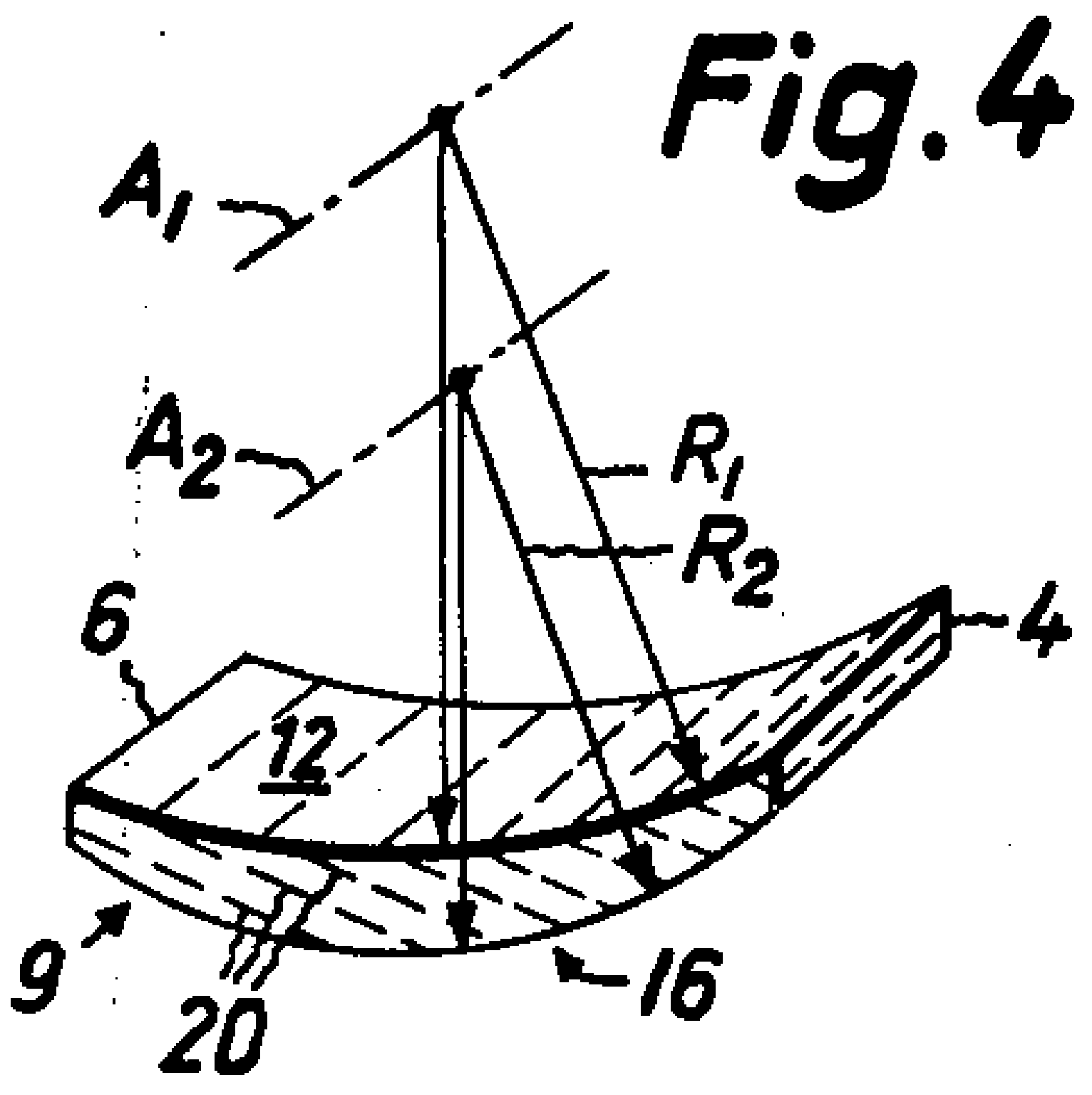


Fig. 5

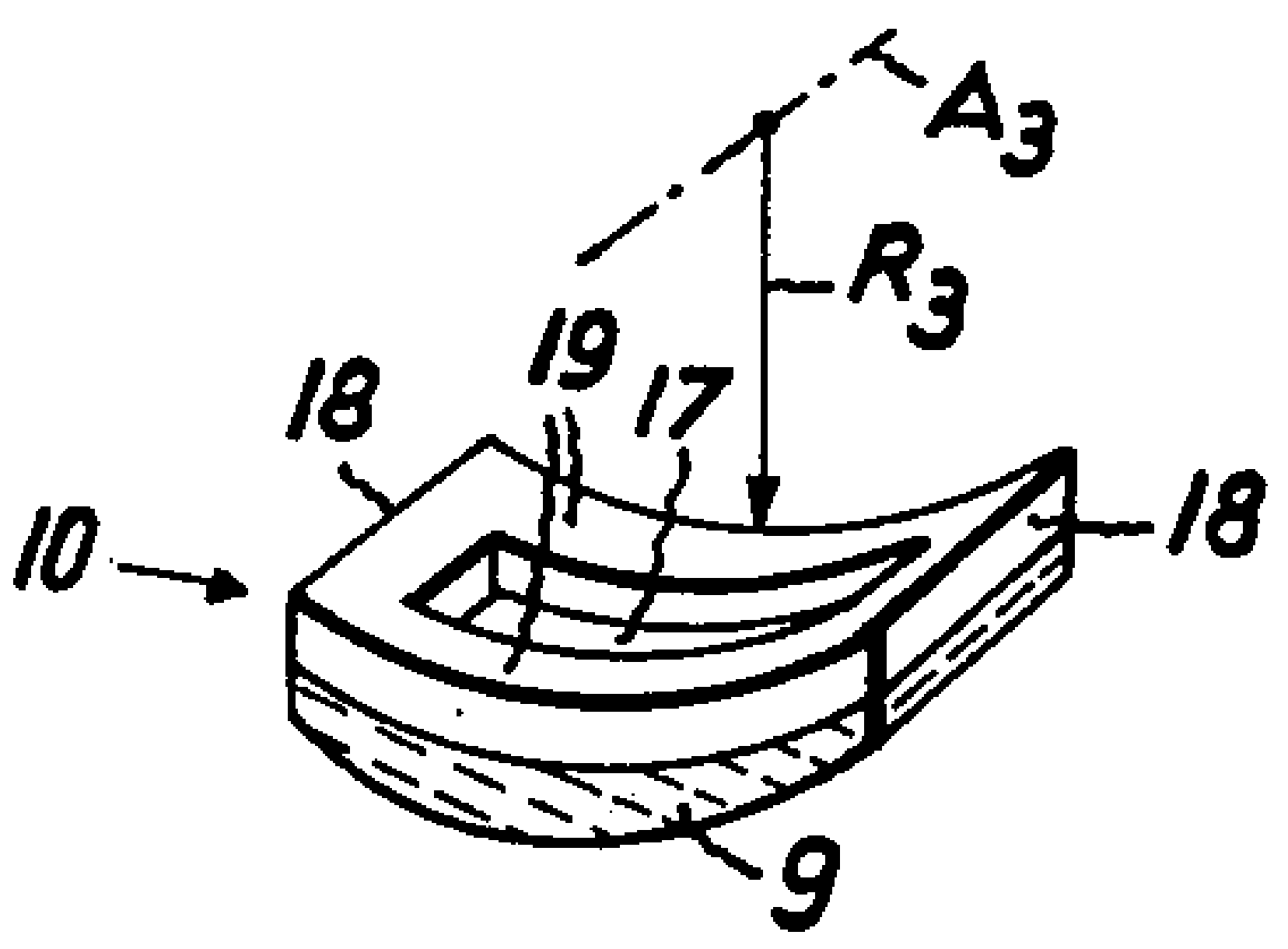


Fig. 6

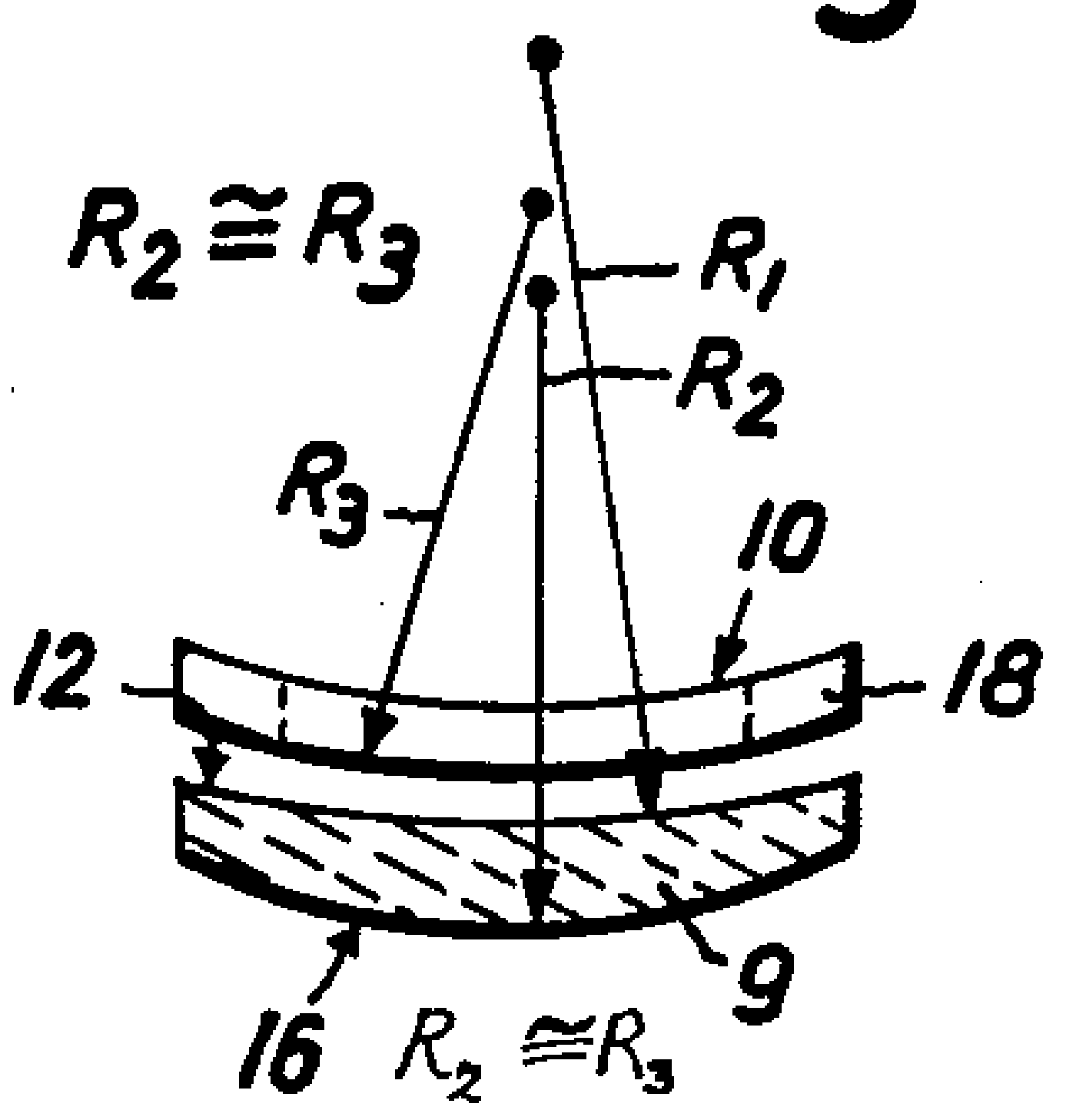
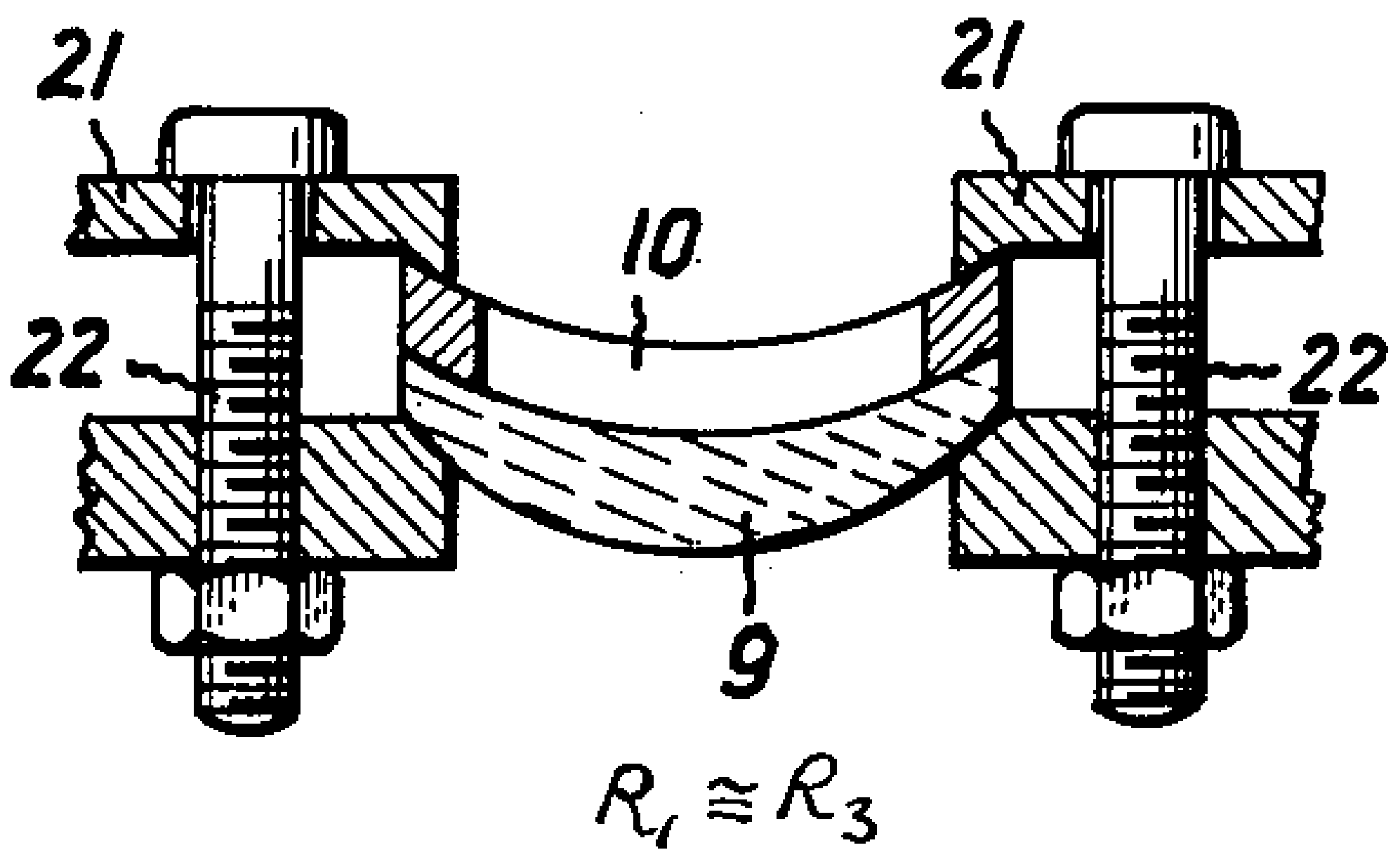


Fig. 7



Inventors

ROLF HOSEMANN
ANTON WEBER

By Taulmin & Taulmin
Attorneys

United States Patent Office

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X-RAY REFRACTING OPTICAL ELEMENT

Rolf Hosemann and Anton Weber, Berlin, Germany, assignors to Licentia Patent-Verwaltungs-G.m.b.H., Hamburg, Germany

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1 Claim. (Cl. 250—51.5)

This invention relates to X-ray refracting elements, and, more particularly, to such X-ray refracting elements which can be used as monochromators in X-ray diffractometry, and as analyzers in X-ray spectroscopy.

By X-rays suitable for the purposes of this invention, we mean electro-magnetic oscillations having wave lengths between approximately 4 and 0.01 Angstroms, which comprise so called gamma rays.

By "refraction," we mean the bending of X-rays by refracting elements at all angles including angles larger than the so-called critical angle which special type of refraction is conventionally called "reflection."

It is an object of our invention to provide an X-ray refracting element of satisfactory size adapted for use as an X-ray monochromator or X-ray analyzer which is capable of producing X-ray images of higher light intensity and freer from distortion than the known crystals.

It is a further object of the invention to provide an X-ray refracting element having the aforesaid properties which is particularly easy to manufacture on an industrial scale.

It is yet another object of the invention to provide X-ray refracting elements whose optical properties such as the intensity of the reflected or refracted X-rays, their freedom from distortion, a sharp selectivity with regard to the refracted frequency band, and the like, can be determined selectively, at will, within certain limits in order to adapt them to a given purpose.

It is well known to use suitable crystals as monochromators or analyzers in X-ray diffractometry and X-ray spectroscopy by interposing these crystals in the path of the X-ray beam.

If these crystals are to be used as monochromators in X-ray diffractometry, they are interposed in the path of the X-ray tube and the preparation so as to filter the beam and obtain a monochromatic X-ray "light" capable of producing a clear diffraction image or pattern. If these crystals are to be used as analyzers in X-ray spectroscopy, they are interposed between the preparation and the X-ray detector and are rotated or displaced in a suitable manner together with the latter so as to disperse spectrally the characteristic radiation emitted by the preparation under examination.

It is desirable to obtain X-ray "images," i.e. diffraction patterns and X-ray spectra which are of high light intensity and free from distortions. This can only be achieved if the aforesaid crystal element has the shape of a plate or disk of largest possible size and consists of a single well-grown crystal. In practice, quartz plates have been used for this purpose which were cut from specimens of outstanding quality selected from among quartz crystals found in nature. Such crystals are relatively rare. Therefore, crystals of various materials such as sodium chloride, calcium fluorite, lithium fluorite, sugar, mica or pentaerythrite, have been grown artificially. However, neither the crystals found in nature nor those artificially grown do usually possess, except in very rare cases, the necessary high quality of their internal texture, if they are of sufficient size; or, if they satisfy the necessary high demands of quality, they can only be found in small-sized specimens.

Furthermore, it was hitherto believed in the art that the aforesaid crystals were particularly well suited for use as X-ray refracting elements, if they possess, particularly in the layers adjacent their refracting surface, a mosaic or tesselated structure. Such structure is brought about in all of the aforesaid crystals by the work process applied in giving the crystal a desired shape. The changes effected especially in those lattice planes of the crystals which are adjacent the refracting surface to give the same a mosaic structure, are also furthered by the heating effects occurring during the working steps of shaping the crystal. These working steps comprise conventionally, for instance, the application of a concave reflecting surface on one side of the crystal, and often the application of a convex side on the crystal opposite the concave one.

In contrast to the hitherto held belief that crystals having at least in their surface a mosaic structure are best sutied for use as X-ray monochromators and analyzers, we have discovered that crystals are much better suited for use as such X-ray refracting elements, and have considerably improved optical properties which comprise a higher intensity of the refracted or reflected X-ray "light," a greater freedom from distortion, and a sharper selectivity of the refracted or reflected frequency bands, as these crystals approach, at least in the layers adjacent the refracting surface, the state of an ideal crystal.

An ideal crystal is a theoretical crystal which is free from any distortions in its crystal lattice.

On the basis of our discovery, the aforesaid objects can be obtained by using as an X-ray refracting element such as a monochromator or analyzer for X-rays having wave lengths from about 4 to 0.01 Angstroms, an artificially grown monocrystal of an elementary semiconductor material selected from the group consisting of germanium and silicon and being shaped or cut as a plate of satisfactory size, which is, at the same time, capable of producing X-ray diffraction patterns or spectra of satisfactory intensity and free from distortion.

We have found that these elementary semi-conductor monocrystals show outstanding mechanical and optical properties which make them superior to all hitherto known crystals. Thus, quartz and all other above mentioned crystals are inclined to adopt a mosaic structure during the mechanical treatment giving the crystal its desired shape. Monocrystals of the semi-conductor elements of germanium and silicon possess a much better thermal conductivity and can therefore be worked to give them the desired shape in accordance with the present invention, while preserving their almost ideal structure, and, as a consequence thereof, their superior optical properties.

On the other hand, it is possible, by a selective mechanical treatment, to bring about a determined degree of recrystallization from an almost ideal structure of at least the lattice portion adjacent the refracting surface of the monocrystal to a partial or complete mosaic structure. By choosing, through a suitable mechanical treatment of the crystal in giving the same its desired shape, a determined stage intermediate the two extremes of complete ideal structure and complete mosaic structure, it is possible, in accordance with our invention, to provide monochromator or analyzer crystals of determined optical behavior.

Both germanium and silicon monocrystals possess reflecting lattice planes (111) which reflect X-rays including Gamma rays with excellent intensity.

If particularly "hard" X-rays are to be reflected, i.e. X-rays having a wave length from 0.01 to about 1 Angstrom, higher indexed lattice planes of germanium or silicon monocrystals may also be used. In the case of germanium, for instance, the relative intensities of X-ray beams reflected by the higher indexed planes compared with the intensity of an X-ray beam reflected by the (111) plane set at 1.0, are 0.9 for the (220) plane, 0.8 for the (131) plane, and 0.7 for the (242) plane.

The absolute intensities or powers of the reflected X-ray beams depend on a considerable number of factors, and their determination is well known in the art. (See, for instance, "Advances in Biological and Medical Physics" by John H. Lawrence and C. A. Tobias, vol. III, page 256 and ff., published by Academic Press, Inc., 1953, New York, N.Y.)

Satisfactory intensity of the reflected X-radiation and freedom from distortion of the resulting X-ray "images" depend to a large degree on the external shape of the crystals used as X-ray refracting elements according to the invention. Crystal plates of the proper shape are obtained by careful cutting, grinding, milling, corroding off, and polishing the artificially grown germanium or silicon crystal.

According to an important feature of our invention, an X-ray refracting element of particularly satisfactory properties is obtained by providing a combined monocrystal plate and convex-concave top frame system in which the refractory surface of the monocrystal is first cut with a determined curvature to be of concave shape and is assembled under pressure to the convex side of the aforesaid frame which convex side is of a narrower curvature than the concave crystal surface, so that the concave monocrystal surface is under permanent stress.

The lattice planes in the monocrystal can be disposed parallel to the refracting or reflecting surface of the monocrystal, or they can be inclined at an acute angle thereto. This angle should not exceed the Bragg reflection angle which is half the diffraction reflection angle. For germanium, this angle is 12° 50′ at the (111) lattice plane, and 22° 50′ at the (220) lattice plane.

Whenever the lattice planes which reflect X-rays are inclined at an angle to the refracting surface of the monocrystal plate, the normal on the group of lattice planes must at least be perpendicular to the shorter of the edges delimiting the refracting surface of the plate.

The shorter edge or edges of that plate surface must be of a length which is above the half value band of the depth of penetration of the X-radiation in the crystal. Otherwise too large a portion of X-ray passes through the crystal without being diffracted. The half value band is in the order of 0.1 micron for the ideal crystal and in the order of 1 micron for a crystal having complete mosaic structure in its near-surface layers.

According to another feature of the invention, that edge of a crystal having a determined different height, length, and width, which corresponds to a length intermediate that of the shortest and the longest edge, is at right angle to the normal on the lattice planes of the monocrystal lattice. If the crystal is so dimensioned, it permits to utilize fully the space angle of the radiation intensity emitted by the X-ray tube, in particular, if line focus tubes are used.

Higher indexed lattice planes of germanium and silicon monocrystals can be used for X-rays having wave lengths from 0.01 Angstrom to $\lambda_{max}=2d$, wherein d is the distance between two higher indexed lattice planes. The use of these higher indexed lattice planes leads to a much higher dispersion, i.e. a small change in the wave length causes a much larger change in the scattering angle than with the (111) plane.

Our invention will be still better understood from the description thereof following hereinafter in connection with the accompanying drawings.

FIGURE 1 is a perspective view of a monocrystal plate according to the invention;

FIGURE 2 illustrates in perspective another embodiment of a monocrystal plate according to the invention;

FIGURE 3 shows in perspective yet a further X-ray refracting element according to the invention, one of the surfaces of which is a portion of the wall of the cylinder;

FIGURE 4 is a further embodiment of a monocrystal X-ray refracting element in perspective view, two opposed surfaces of which are portions of the walls of cylinders having different generating radii;

FIGURE 5 illustrates in perspective an X-ray refracting system comprising a monocrystal of the kind illustrated in FIGURE 4 filled onto a curved frame;

FIGURE 6 is a lateral view of the parts of the system shown in FIGURE 5 prior to its assembly; and,

FIGURE 7 is a lateral view in cross section of the assembled system.

Referring now to the drawings in detail: the X-ray refracting plate **1** illustrated in FIGURE 1 has the height **4**, the length **5**, and the width **6**, all three being of different length. The plate is a rectangular parallelepiped, its edges **4**, **5**, and **6**, being straight lined and at right angle to each other. The lattice planes **2** are indicated by dashed lines, and the normal **3** on these lattice planes of the monocrystal is also normal to the largest end surface **11** of plate **1**.

FIGURE 2 shows a monocrystal X-ray refracting element identical with that shown in FIGURE 1 and like references numerals indicating like parts. However, the lattice planes **20** are inclined relative to the largest end surface **11** of the monocrystal plate **1**, forming an acute angle α with that surface **11**. Consequently, the normal **3** on the group of lattice planes **20** is inclined at that same angle α to the normal **7** on plane **11**, for instance, at the edge **6** which is of a length intermediate the lengths of edge **4** being the shortest, and of edge **5** being the longest of the three edges, for reasons set forth above. The angle α is smaller than the Bragg reflection angle.

In FIGURE 3 the X-ray refracting element **8** has two opposed largest end surfaces **12** and **13**, of which surface **12** is concavely shaped to form a portion of the wall of a cylinder. Surface **13** opposite surface **12** is planar. Again, the monocrystal plate has four shortest edges **4**, four edges **6** of the medium length, and two straight bottom edges **14** delimiting surface **13**, and two curved top edges **15** delimiting cylindrical surfaces. Again, as in FIGURE 2, the lattice planes **20** are inclined at an angle relative to the end surfaces **12** and **13** in such a manner that the normal **3** on the group of lattice planes **20** forms an angle β with the normal **7** on plane **13**, for instance, at the edge **6** of medium length.

The axis forming the central axis of the cylinder having the generating radius R is preferably parallel to that medium long edge **6**. The curved surface **12** can be ground into the monocrystal.

The embodiment of the X-ray refracting element of our invention shown in FIGURE 4, which element is designated by reference numeral **9**, is similar to that shown in FIGURE 3 inasmuch as it possesses shortest edges **4**, medium long edges **6**, and curved edges **15** delimiting one curved end surface **12** which forms a portion of the wall of a cylinder having the central axis A_1 and a generating radius R_1, while the surface **16** opposite concave surface **12** is convex and forms a portion of the wall of a cylinder having the central axis A_2 and the generating radius R_2. Cylinder axes A_1 and A_2 are located in the same plane being the central plane of the X-ray refracting element **9** and parallel to the end surfaces delimited by edges **4** and **6**. Radius R_1 is longer than radius R_2, and axes A_1 and A_2 are both parallel to medium long edges **6** of element **9**.

The X-ray refracting system illustrating in FIGURE 5 is obtained from the parts thereof shown in FIGURE 6. One of these parts is a monocrystal element **9** as illustrated in FIGURE 4. The other part is a frame **10** having a central window **17** having parallel marginal portions **18** opposite each other the length of which corresponds to the length of edges **6** of element **9**. Frame portions **19** interconnecting portions **18**, are curved in a plane extending in the wall of a cylinder having the generating radius R_3 which is smaller than radius R_1 pertaining to surface **12** of element **9**, while cylinder axis A_3 is parallel to cylinder axes A_1 and A_2.

Frame **10** is placed with the convex side of curved frame portions **19** on the surface **12** of monocrystal element **9** and is pressed thereagainst, for instance, by clamping means **21** in such a manner that monocrystal element **9** is bent under stress so that surface **12** adopts the same curvature as frame portions **19**, corresponding to radius R_3.

Radius R_3 is preferably equal to radius R_2 pertaining to convex surface **16** of element **9**. In the assembled system, the surfaces **12** and **16** of element **9** thus extend approximately parallel to each other at least in the central portion of element **9** facing the window **17** of frame **10**.

By adjusting the tension of clamping means **21**, for instance, with the aid of screw means **22**, it is possible to vary the curvature of the concave surface **12** of monocrystal **9** somewhat, and thereby to vary the angle of the lattice planes relative to an incident X-ray beam.

It will be understood that this invention is susceptible to modification in order to adapt it to different usages and conditions, and, accordingly, it is desired to comprehend such modifications within this invention as may fall within the scope of the appended claim.

We claim:

An X-ray refracting element for X-rays having wave length from 0.01 to about 4 Angstroms consisting of an artificially grown monocrystal of an elementary semiconductor material selected from the group consisting of germanium and silicon, said monocrystal being shaped as a plate having planar surface and shorter and longer lateral edges thereof, and comprising a group of X-ray reflecting lattice planes, said lattice planes forming an angle smaller than half the diffraction angle with said plate, the normal of said group of lattice planes being disposed at a right angle to the shorter lateral edges of said plate.

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