Sept. 3, 1957 A. R. LANG 2,805,342

DIFFRACTOMETER

Filed July 12, 1954

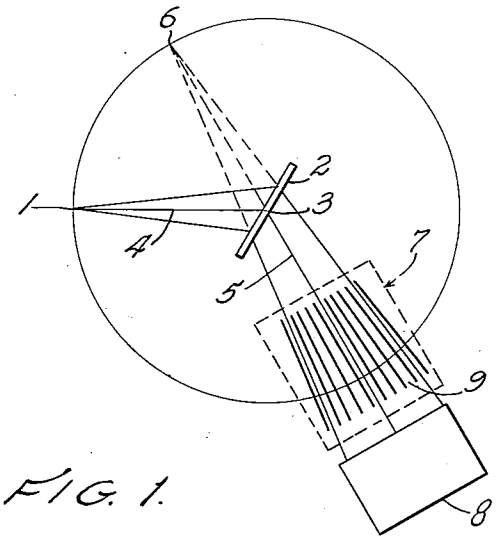


FIG. 1.

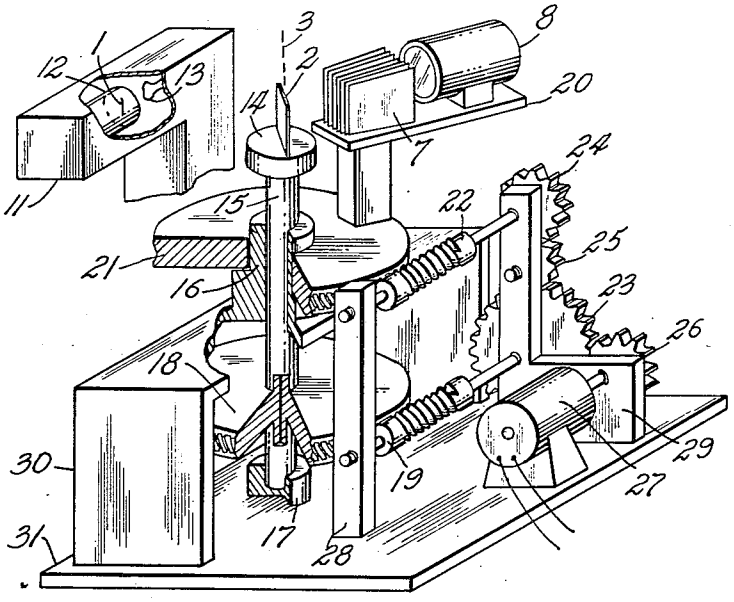


FIG. 2.

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2,805,342

Patented Sept. 3, 1957

2,805,342

DIFFRACTOMETER

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Application July 12, 1954, Serial No. 442,567

5 Claims. (Cl. 250—53)

The present invention relates to diffractometers.

Specimens are commonly analyzed with the aid of X-rays that are caused to impinge thereon in the form of a diverging primary beam that is reflected therefrom by diffraction in the form of a converging beam that is thereafter detected. The specimen is mounted upon a rotatably adjustable support.

In many applications, however, as where it is necessary to study diffracted rays scattered at small angles, and where it is desirable to study preferred orientation textures of the material of the specimen, improved results are obtained by transmitting the primary X-ray beam through the specimen, instead of reflecting it therefrom.

An object of the present invention is to provide a new and improved diffractometer operating on the principle of transmitting the primary X-ray beam through the specimen.

With the above end in view, a feature of the invention resides in positioning a collimator of particular type in the path of the secondary diverging X-ray beam transmitted by diffraction through the specimen. The collimator is provided with a plurality of X-ray transmission channels converging longitudinally toward the virtual focus of the diverging secondary X-ray beam when the collimator occupies a predetermined position. The collimator may be rotatably adjusted about the axis of rotation of the said rotatably adjustable support at double the rate of rotatable adjustment of the specimen about the said axis of rotation to an optimum position for effecting recording of the data.

Other and further objects of the invention will be explained hereinafter and will be particularly pointed out in the appended claims.

The invention will now be more fully described in connection with the accompanying drawings, in which Fig. 1 is a diagrammatic view illustrating geometric principles underlying the present invention; and Fig. 2 is a perspective, partly in section and partly broken away, of a diffractometer embodying the invention.

A primary beam is diagrammatically shown in Fig. 1 diverging from a source 1 of X-rays, illustrated in Fig. 2 as a straight-line source produced by bombarding the target 12 of an X-ray tube 11 by a beam of electrons emitted from a filament 13. The straight-line source 1 is diagrammatically indicated in Fig. 1 as perpendicular to the plane of the drawing, which may represent the scanning plane. The primary X-ray beam is shown in Fig. 1 diverging toward an X-ray diffraction transmission specimen 2 that it is desired to analyze. It is customary to adjust the support for the specimen 2, shown in Fig. 2 at 14, rotatably about an axis 3 substantially perpendicular to the primary diverging X-ray beam and parallel to the straight-line X-ray source 1. The center or median ray 4 of the incident primary diverging X-ray beam is therefore shown impinging upon a central part of the specimen 2 at 3, approximately half-way between the extreme rays of the diverging beam. The primary beam is transmitted by diffraction through the specimen 2 in the form of a secondary X-ray beam that is shown diverging from a straight-line virtual focus 6 parallel to the straight-line source 1.

Prior-art diffractometers are not well adapted to the study of a diffracted X-ray beam that makes a small angle with the primary beam, for the following reasons. First, the length of the reflection specimen heretofore used that is irradiated by a primary beam of fixed divergence increases rapidly as the angle made by the primary beam with the specimen surface is reduced. Secondly, the aberrations of the Bragg-Brentano focussing geometry increase rapidly as the observed diffraction angle is reduced. Such increased aberration results in loss of angular resolution and reduction in the accuracy of determination of the diffracted angle. It is essential to record accurately diffracted small angles, however, in the study of important classes of substances, such as fibres, fats, soaps and solutions. It is for this reason that the specimen 2 is illustrated as a transmission specimen.

In a copending application, Serial No. 442,566, filed July 12, 1954, there is disclosed a diffractometer designed to obtain high resolution with the use of an X-ray diffraction reflection specimen disposed in the path of a primary X-ray beam the angle of divergence of which is usually between one and four degrees. This divergent primary X-ray beam becomes reflected from the specimen in the form of an X-ray beam that converges toward a scanning reception slit. Since the diffracted rays transmitted by the diffraction transmission specimen 2 described herein diverge, however, it is not possible to employ a scanning reception slit with a primary X-ray beam of divergence as great as from one to four degrees. It would be necessary to reduce the angle of divergence of the primary X-ray beam and, therefore, also that of the diffracted beam to a very much smaller value. This value would be so small as to be of the same order of magnitude as the angular resolution required in scanning the spectrum, which is usually a fraction of one degree of scattering angle. Such a reduction of primary beam divergence would result in so great a loss of total intensity of diffracted beam as to render it difficult accurately to record the diffraction patterns of materials having low angle diffraction patterns. Many such materials, furthermore, are also weak scatterers of X-rays.

In another class of X-ray diffraction study, it is required to find the preferred orientation of crystallites within a polycrystalline fiber, sheet or foil. In such work, also, it is necessary to use a diffraction transmission specimen in order to determine the distribution of atomic plane normals in the general direction of the planes of the foil or the sheet or the plane containing the fibre axis. Here, too, a reduction of the angle of divergence of the primary beam will result in loss of intensity. There is the additional disadvantage, moreover, that the use of a primary beam of greatly reduced divergence results in a reduction of the number of crystallites contained in the irradiated volume of the specimen. This gives rise to statistical uncertainties in the determination of their orientation distributions.

The defects of the Bragg-Brentano focussing method in the study of beams diffracted at low angles are entirely overcome by the present invention, which provides an apparatus for use with a transmission specimen disposed in a primary X-ray beam of several degrees divergence in the scanning plane. This result is obtained though the diffraction pattern is recorded with a resolution of diffraction angle of a fraction of one degree.

According to a preferred embodiment of the invention, herein illustrated and described, an X-ray collimator 7 is interposed in the path of the diverging secondary X-ray

beam transmitted by diffraction through the specimen 2 in the form of a diverging secondary X-ray beam. A detector 8 is disposed in the path of the X-ray beam transmitted through the collimator.

The collimator 7 is provided with a plurality of X-ray transmission channels 9 converging longitudinally toward the virtual focus 6, when the collimator occupies a predetermined position.

The channels are conveniently constructed from sets of thin partitions with spacers between said partitions. The partitions are conveniently made of thin foil of metal which is strongly absorbing to the X-ray wavelengths used. It is desirable that the ratio of width of each channel to the thickness of each of such partitions be made as large as possible so that the greatest fraction of the radiation in the diffracted beam may be transmitted, hence it is desirable that the partitions be thin. If each channel element is of the same width then the axes of neighboring channels must be inclined to each other by the same small angle. This condition is conveniently satisfied by making either the partitions, or the spacers, or both, in the form of wedges of small taper angle.

It has been found convenient to provide means for adjusting the distance of 7 from the diffractometer axis 3, and also for making a rotational adjustment of the collimator 7 in the scanning plane so that the point of convergence of the axes of the channels may be made accurately coincident with the focus 6.

The collimator 7 is adjustable about the axis of rotation 3. The straight-line source 1 and the virtual focus 6 form line elements of a circular cylinder of which the axis of rotation is shown at 3.

Each channel in the scanning plane is narrow enough and of suitable length to restrict the angular range of X-rays in the scanning plane transmitted by it to a desired small fraction of a degree, which is of the same order of magnitude as the required angular resolution of recording of the diffraction specimen.

The channels are so oriented with respect to one another that each channel transmits rays which have been separately diffracted by the specimen 2 but deviated through the same mean angle.

The number of the channels is sufficiently great, fifty to one hundred, and the angle subtended thereby at the specimen 2 is sufficiently large, to receive diffracted rays coming from the entire illuminated area of the specimen 2 corresponding to the whole divergence of the incident primary X-ray beam.

At any given setting, however, the collimator transmits only rays included in the range of diffraction angles substantially narrower than the divergence of the incident primary X-ray beam.

The support 14 is shown in Fig. 2 mounted on the upper end of a shaft 15 the axis of rotation of which is shown at 3. The lower end of the shaft 15 is mounted in a thrust bearing 17 secured upon a base plate 31. An intermediate portion of the shaft 15 is mounted in a bearing 16 provided upon a housing frame 30 secured to the base plate 31. The X-ray tube 11 is held in fixed position relative to the base plate 31. The shaft 15 is integral with a worm wheel 18 in the housing frame 30. The worm wheel 18 is driven from a worm 19.

The collimator 7 and the detector 8 are mounted upon a scanning arm 20 that is integral with a worm wheel 21 mounted coaxial with the shaft 15. The worm wheel 21 is driven from a worm 22.

The worm wheels 18 and 21 are of the same diameter and have the same number of teeth. The worms 19 and 22, of identical construction, are journaled in plates 28 and 29, rising vertically from the base plate 31.

A gear 26 upon the shaft of a motor 27, mounted on the base plate 31, meshes with a gear 23 upon the shaft of the worm 19. The gear 23, in turn, through an idler 25, drives a gear 24 on the shaft of the worm 22. The diameter of the gear 23 is double that of the gear 24. The gear 23, therefore, has twice as many teeth as the gear 24. The worm wheel 21 and, therefore, the scanning arm 20 are thus rotated, in the same direction of rotation, at double the rate of rotation of the worm wheel 18 and the shaft 15 that is fixed thereto.

The worm wheels 18 and 21 are initially adjusted rotatably to a position such that the angle of incidence of the center or median ray 4 of the primary diverging beam equals the angle of emergence of the center or median ray 5 of the secondary diverging beam transmitted through the specimen 2, as illustrated in Fig. 1. This will ensure that these angles shall be maintained equal in all positions of rotatable adjustment of the worm wheels 18 and 21 effected by the motor 27.

During this rotation of the specimen 2 about the axis 3, the X-rays transmitted through the collimator channels will diverge from the virtual straight-line focus 6. This virtual straight-line focus 6 will be disposed on a circular cylinder coaxial with the axis of rotation 3. The straight-line source 1 is disposed along a line element of this cylinder.

It is because of this construction, whereby the virtual focus 6 is at a constant distance from the axis of rotation 3, that it is possible to adjust the collimator rotatably about the axis 3. The collimator 7 functions as a diffraction-angle selector, operating on all the X-rays transmitted through the specimen 2 substantially independently of the divergence of the primary X-ray beam.

Modifications may be made by persons skilled in the art and all such are considered to fall within the spirit and scope of the invention as defined in the appended claims.

What is claimed is:

1. An X-ray diffractometer comprising means for supporting in the path of a primary X-ray beam diverging from a source of X-rays an X-ray diffraction transmission specimen in order that the X-rays may be transmitted therethrough by diffraction in the form of a diverging secondary X-ray beam having a virtual focus, and a collimator disposed in the path of the diverging secondary X-ray beam provided with a plurality of X-ray transmission channels converging longitudinally toward the said virtual focus when the collimator occupies a predetermined position, and a detector disposed in the path of the X-rays transmitted through the transmission channels.

2. An X-ray diffractometer comprising means for supporting in the path of a primary X-ray beam diverging from a source of X-rays an X-ray diffraction transmission specimen in order that the X-rays may be transmitted therethrough by diffraction in the form of a diverging secondary X-ray beam having a virtual focus, the supporting means being rotatably adjustable about an axis substantially perpendicular to the primary beam, and a collimator adjustable in the path of the diverging secondary X-ray beam along the surface of a circular cylinder coaxial with the said axis of rotation, the collimator being provided with a plurality of X-ray transmission channels converging longitudinally toward the said virtual focus when the collimator occupies a predetermined position of adjustment, and a detector disposed in the path of the X-rays transmitted through the transmission channels.

3. An X-ray diffractometer comprising means for supporting in the path of a primary X-ray beam diverging from a source of X-rays an X-ray diffraction transmission specimen in order that the X-rays may be transmitted therethrough by diffraction in the form of a diverging secondary X-ray beam having a virtual focus, the supporting means being rotatably adjustable about an axis substantially perpendicular to the primary beam, and a collimator adjustable in the path of the diverging secondary X-ray beam along the surface of a circular cylinder coaxial with the said axis of rotation, the collimator being provided with a plurality of X-ray trans-

mission channels converging longitudinally toward the said virtual focus when the collimator occupies a predetermined position of adjustment, a detector disposed in the path of the X-rays transmitted through the transmission channels, and means for rotatably adjusting the collimator about the said axis of rotation at double the rate of rotatable adjustment of the specimen about the said axis of rotation.

4. An X-ray diffractometer comprising means for supporting in the path of a primary X-ray beam diverging from a source of X-rays an X-ray diffraction transmission specimen in order that the X-rays may be transmitted therethrough by diffraction in the form of a diverging secondary X-ray beam having a virtual focus, the supporting means being rotatably adjustable about an axis substantially perpendicular to the primary beam, and a collimator adjustable in the path of the diverging secondary X-ray beam along the surface of a circular cylinder coaxial with the said axis of rotation, the collimator being provided with a plurality of X-ray transmission channels converging longitudinally toward the said virtual focus when the collimator occupies a predetermined position of adjustment, and a detector disposed in the path of the X-rays transmitted through the transmission channels, the channels being of length and width to transmit X-rays of the diverging secondary X-ray beam over a narrow range of diffraction angles substantially determinative of the angular resolution of recording of the diffraction pattern.

5. An X-ray diffractometer comprising means for supporting in the path of a primary X-ray beam diverging from a source of X-rays an X-ray diffraction transmission specimen in order that the X-rays may be transmitted therethrough by diffraction in the form of a diverging secondary X-ray beam having a virtual focus, the supporting means being rotatably adjustable about an axis substantially perpendicular to the primary beam, and a collimator adjustable in the path of the diverging secondary X-ray beam along the surface of a circular cylinder

coaxial with the said axis of rotation, the collimator being provided with a plurality of X-ray transmission channels converging longitudinally toward the said virtual focus when the collimator occupies a predetermined position of adjustment, the channels being disposed to transmit X-rays with the same mean diffraction angle, a detector disposed in the path of the X-rays transmitted through the transmission channels, the channels being of length and width to transmit X-rays of the diverging secondary X-ray beam over a narrow range of diffraction angles substantially determinative of the angular resolution of recording of the diffraction pattern, the number of the channels being sufficiently great to transmit all the X-rays within the entire range of divergence of the X-rays of the primary beam that are diffracted at the said mean diffraction angle, and means for rotatably adjusting the collimator about the said axis of rotation at double the rate of rotatable adjustment of the specimen about said axis of rotation, the initial angular position of the specimen being so adjusted about the said axis of rotation that, during the rotation of the specimen, the median ray in the primary X-ray beam and the median ray in the secondary X-ray beam shall be equally inclined thereto.

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