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DIFFRACTOMETER

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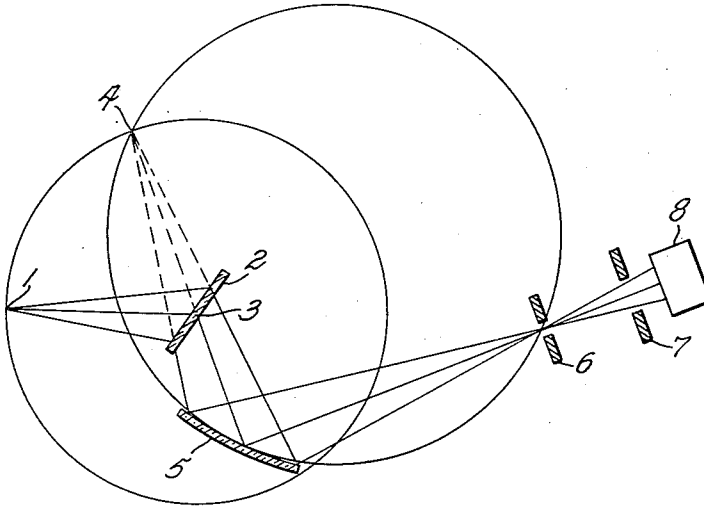


FIG. 1.

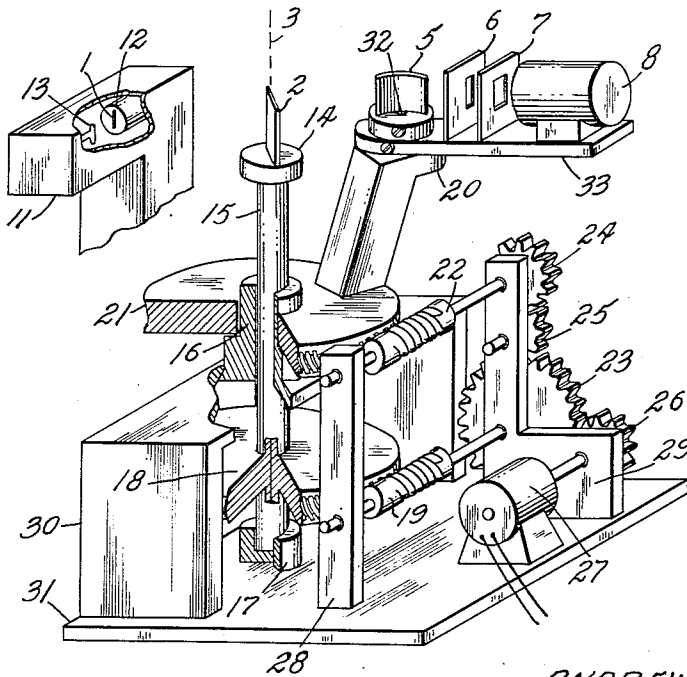


FIG. 2.

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DIFFRACTOMETER

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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 the provision of a reflection crystal monochromator from which is reflected by diffraction the secondary diverging X-ray beam transmitted through the specimen in the form of a converging X-ray beam. The converging X-ray beam is transmitted through a detection slit disposed at the focus of this converging beam. The monochromator crystal, the detection slit and the detector are rotatably adjustable as a unit 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 the 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 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. 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 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

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a straight-line virtual focus 4 parallel to the straight-line source 1.

The quality of the diffraction pattern obtained is improved with the diffractometer of the present invention.

This will be understood from the following considerations. The straight-line source 1 of X-rays is shown in Fig. 2 produced by bombarding the target 12 of an X-ray tube 11 by a beam of electrons emitted from a filament 13. The X-rays so emitted, however, are not confined to only the single wavelength X-radiations characteristic of the tube target 12 which are diffracted by the specimens according to Bragg's Law. The X-rays so emitted and diffracted by the specimen 2 cover a continuous range of wavelengths, known as the "white" radiation. A continuous "white"-radiation background is thus produced which tends to obscure the relatively weak characteristic diffraction pattern, thereby reducing the precision of measurement of the intensity of even the strongest diffracted characteristic rays. The undesirable effect of the background is increased by the fluorescent radiation excited by the primary divergent X-ray beam, impinging upon the specimen 2, by its spontaneous radioactivity, and by the so-called Compton incoherent scattering of the characteristic radiation in which a small change in wavelength is produced.

In accordance with the present invention, a reflection crystal monochromator 5 is disposed in the path of the diverging secondary X-ray beam. A barrier 6 is provided with a straight-line slit parallel to the axis of rotation 3, disposed at the straight-line focus of convergence of the converging X-ray beam reflected by diffraction of the diverging secondary X-ray beam from the crystal monochromator 5. For definiteness, the slit in the barrier 6 will hereinafter be referred to as a detection slit. A detector 8 is disposed in the path of the X-ray beam transmitted through the detection slit. A further barrier 7 provided with a straight-line slit parallel to the axis of rotation 3 is positioned either before or after the barrier 6, the slit being wide enough to pass a beam of the degree of divergence required but no wider. The crystal monochromator 5 is shaped to curve its lattice planes concavely in the form of concentric circular cylinders with axes parallel to the said axis of rotation 3.

The focusing conditions of Johann or Johansson may be obtained by suitably spacing the crystal monochromator 5 from the virtual focus 4 and the barrier 6. The aperture of the detection slit in the barrier 6 may be adjusted to adjust the range of wavelengths, centered on any required wavelength, that are finally received by the detector 8.

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 barriers 6 and 7, the crystal monochromator 5 and the detector 9 are mounted upon a scanning arm 20 that is integral with a worm wheel 21 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 of the primary diverging beam equals the angle of emergence of the center or median ray 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, and the virtual straight-line focus 4 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 may be necessary to readjust the positions of the crystal monochromator 5, the barriers 6 and 7 and the detector 8 when different monochromator crystals 5 are employed. To this end, the monochromator crystal 5, the barriers 6 and 7 and the detector 8 are shown in Fig. 2 mounted upon an extension arm 33 that is adjustably pivoted about a pivot 32 of the scanning arm 20. The monochromator 5 and the barriers 6 and 7, as well as the apertures of the slits thereof, are adjusted until the detector 8 records the highest intensity for the degree of monochromatization desired in the particular experiment. The extension arm 33 is fixed in adjusted position by a set screw, and the monochromator 6 is fixed in adjusted position by another set screw. The set screws bear upon the common pivot 32 about which the monochromator 6 and the extension arm 33 are pivotally adjustable.

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, the supporting means being rotatably adjustable about an axis substantially perpendicular to the primary beam, a reflection crystal monochromator disposed in the path of the diverging secondary X-ray beam for reflecting the diverging secondary X-ray beam into a beam of X-rays converging to a second focus, a barrier having a detection slit disposed at the second focus, and a detector disposed in the path of the X-ray beam transmitted through the detection slit.

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-ray 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, there being disposed in the path of the diverging secondary X-ray beam a reflection crystal monochromator the lattice planes of which are concavely curved in the form of concentric circular cylinders with axes parallel to the said axis of rotation, the radius of curvature of the concavely curved lattice planes being chosen so that predetermined X-ray wavelengths in the diverging secondary X-ray beam are reflected by it to form a converging beam, a barrier having a straight-line detection slit disposed parallel to said axis of rotation at the straight-line focus of convergence of the converging X-ray beam reflected from the said concavely curved lattice planes, and a detector disposed

in the path of the X-ray beam transmitted through the detection slit.

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, there being disposed in the path of the diverging secondary X-ray beam a reflection crystal monochromator the lattice planes of which are concavely curved in the form of concentric circular cylinders with axes parallel to the said axis of rotation, the radius of curvature of the concavely curved lattice planes being chosen so that predetermined X-ray wavelengths in the diverging secondary X-ray beam are reflected by it to form a converging beam, a barrier having a straight-line detection slit disposed parallel to said axis of rotation at the straight-line focus of convergence of the converging X-ray beam reflected from the said concavely curved lattice planes, a detector disposed in the path of the X-ray beam transmitted through the detection slit, and means for rotatably adjusting the crystal monochromator, the barrier and the detector as a unit 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 and 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, there being disposed in the path of the diverging secondary X-ray beam a reflection crystal monochromator the lattice planes of which are concavely curved in the form of concentric circular cylinders with axes parallel to the said axis of rotation, the radius of curvature of the concavely curved lattice planes being chosen so that predetermined X-ray wavelengths in the diverging secondary X-ray beam are reflected by it to form a converging beam, a barrier having a straight-line detection slit disposed parallel to said axis of rotation at the straight-line focus of convergence of the converging X-ray beam reflected from the said concavely curved beam transmitted through the detection slit, and means for rotatably adjusting the crystal monochromator, the barrier and the detector as a unit about the said axis of rotation at double the rate of rotatable adjustment of the specimen about the 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.

References Cited in the file of this patent

UNITED STATES PATENTS

2,452,045	Friedman	Oct. 26, 1948
2,474,835	Friedman	July 5, 1949
2,511,152	Ekstein	June 13, 1950
2,532,810	Harker	Dec. 5, 1950
2,540,821	Harker	Feb. 6, 1951

OTHER REFERENCES

- Focusing X-ray Monochromators, C. S. Smith, Review of Scientific Instruments, June 1941, pp. 312, 313, 314.
 Diffracted-Beam Monochromatization Techniques in X-Ray Diffractometry, A. R. Lang, Review of Scientific Instruments, January 1956, page 17.