Feb. 26, 1957 S. WYTZES 2,783,385

X-RAY FLUORESCENT SPECTROMETRY

Filed Oct. 30, 1953 2 Sheets-Sheet 1

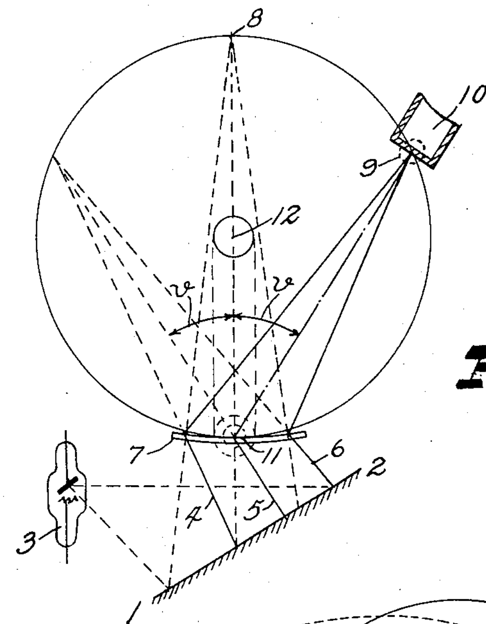

Fig. 1

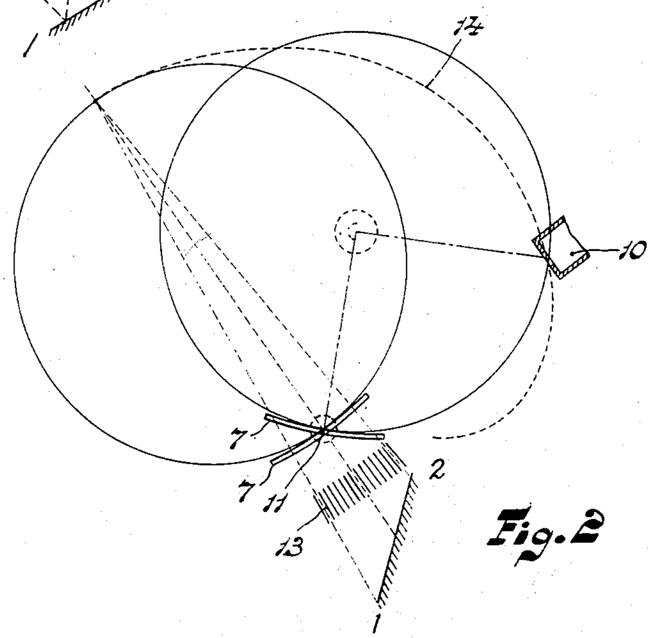

Fig. 2

INVENTOR
SJOERD WIJTZES

BY Fred M Vogel
AGENT

Feb. 26, 1957 S. WYTZES 2,783,385

X-RAY FLUORESCENT SPECTROMETRY

Filed Oct. 30, 1953 2 Sheets-Sheet 2

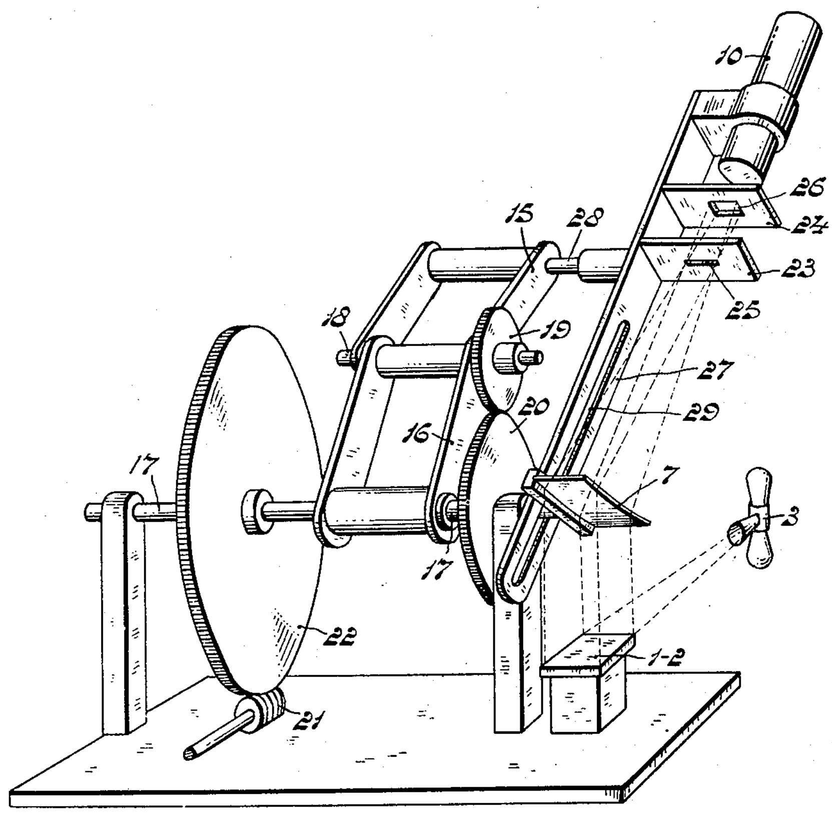

Fig. 3

INVENTOR

SJOERD WIJTZES

BY Fred M Vogel

AGENT

United States Patent Office

2,783,385

Patented Feb. 26, 1957

1

2,783,385

X-RAY FLUORESCENT SPECTROMETRY

Sjoerd Wytzes, Eindhoven, Netherlands, assignor to Hartford National Bank and Trust Company, Hartford, Conn., as trustee

Application October 30, 1953, Serial No. 389,448

Claims priority, application Netherlands November 8, 1952

6 Claims. (Cl. 250—53)

This invention relates to an X-ray fluorescent analysis apparatus employing a focussing crystal.

Compounds of materials may be examined by X-ray techniques by exposing the same to X-ray so as to excite the elements thereof into a condition where they emit secondary X-rays having wavelengths which are characteristic of the nature of the elements. Thus, a poly-chromatic radiation is obtained which is characteristic of the material concerned. By measuring the wavelengths of this radiation and the associated intensities, the elements of the material and their proportions can be determined.

For separating X-rays of equal wavelength from the poly-chromatic radiation, a single crystal plate may be employed in which the lattice planes are parallel or substantially parallel to the surface. The rays emitted by the material strike the crystal, and if the angle of incidence of the rays at the lattice planes corresponds to the so-called Bragg angle for a wavelength of the mixture of rays, reflection takes place and a beam of monochromatic X-rays of this wavelength is produced. The intensity is measured with the aid of a detector responsive to X-rays and the wavelength is derived from the angle of reflection between the deflected beam and the lattice planes of the crystal by the formula $n\lambda=2d \sin \theta$, wherein d designates the distance between the lattice planes and θ the aforesaid angle. Alternatively, a transmission crystal plate may be employed, in which the lattice planes are at right angles or substantially at right angles to the surface, this plate being positioned relative to the rays emitted by the material such that the rays pass through the crystal. The lattice planes deflect only rays of a definite wavelength, which strike the lattice planes at an angle at which the aforesaid Bragg formula is fulfilled.

A known apparatus of the foregoing type employs a focussing or curved single crystal for separating and increasing the intensity of the various component wavelengths of the poly-chromatic fluorescent radiation. In this apparatus, the X-ray responsive detector is positioned along the arc of a circle, the center of which lies in the crystal surface, and is rotatable about that center along the arc of the circle. At the same time, the focussing crystal is rotatable about an axis through this center, parallel to the lattice planes and at right angles to the plane of the circle, the angular speed of the rotation of the crystal being half the angular speed of the detector. A collimator is also provided between the material to be examined and the single crystal, so that direct rays are prevented from striking the detector.

This known apparatus, however, suffers from the drawback that the arc along which the detector is moved does not correspond to those positions at which the smallest sectional area, i. e. the focal spot, of the focussed beam of rays deflected by the crystal is located. To obviate this, the known apparatus provides for continuously changing the radius of curvature of the focussing crystal for each position of the X-rays detector which necessitates provision of a cumbersome, expensive, and inaccurate device.

2

The chief object of the present invention is to provide an apparatus of the foregoing type which eliminates the need for providing means for varying the curvature of the single focussing crystal as a function of the position of the detector.

In an apparatus according to the invention, the X-rays falling at different angles on the curved crystal and deflected by reflection from the lattice planes will, in principle, converge to form line shaped images having cross sections located on the circumference of a circle defined by the curved crystal surface and having a diameter equal to the radius of curvature of the crystal, which will be referred to hereinafter as the measuring circle. According to the invention, the detector is arranged to be rotatable about the center of this measuring circle.

Upon rotation of the curved crystal about its axis in the concave surface, which axis is at right angles to the plane of the measuring circle, the focussed X-ray spots will define a kind of cycloidal path. Means are provided so that rotation of the detector about the center of the measuring circle will cause it to follow the same cycloidal path and, thus, always occupy the optimum position for receiving the X-rays, which involves rotating the detector with twice the angular speed of the crystal about its point of rotation, which moves with the crystal.

One embodiment of the apparatus according to the invention, comprises a curved transmission crystal which is rotatable about a rotary shaft provided with an arm secured rigidly thereto having an extension pivotable about the free end of the arm. The opposite end of the extension supports the detector, which is also guided by a slotted member which does not move with the rotation of the crystal. The arm and extension have dimensions and are linked so that upon rotation of the crystal shaft, the angle between the arm and the extension is always double the angle through which the crystal has rotated. For this purpose, the extension may be provided with a toothed wheel secured thereto and centered relative to the free end of the arm and engaging a stationary circular toothed rack, which is centered relative to the shaft about which the crystal rotates, the pitch circle of this toothed rack having twice the diameter of the toothed wheel. Alternatively, in place of the toothed rack, use may be made of a combination of rope discs. The detector is rotatably secured to the extension and is provided with a directional arm, which remains always orientated towards the rotary shaft of the crystal so that where the detector responds only to rays incident in a definite direction, the central rays of the deflected beams will be deflected in that direction.

The invention will now be described with reference to the accompanying drawing in which:

Fig. 1 illustrates the principle of operation of the apparatus according to the invention;

Fig. 2 shows the cycloidal path described by the detector;

Fig. 3 shows diagrammatically one structural embodiment of an apparatus according to the invention.

Referring now to Fig. 1, the line **1—2** designates the surface of a specimen material to be examined which is irradiated by X-radiation from an X-ray tube **3**. Under the action of the incident or primary X-rays, the elements of the material are excited; whereupon the surface of the specimen **1—2** becomes a source of secondary X-rays and emits a poly-chromatic radiation of wavelengths which are characteristic of the substances contained in the material.

A focussing crystal in the shape of a thin plate **7**, for example, of quartz or mica, is positioned in the path of the secondary radiation. This crystal has lattice planes lying in the direction of thickness of the plate. The initially flat plate is curved in a known manner such that these lattice planes are directed towards a point 8. The point 8 and the curved crystal define a circle, the center of which is designated 12, having a radius approximately equal to half the radius of curvature of the inner side of the curved crystal plate 7. This circle has already been referred to as the "measuring circle."

Rays striking the lattice planes directioned to the point 8 under an angle θ, for example, the rays 4, 5 and 6 shown, are deflected by the crystal if their wavelength fulfills the Bragg equation $n\lambda=2d \sin \theta$, wherein d designates the distance between the lattice planes. Whenever these rays strike the lattice planes under the same angle at points lying on the same circle going through the point 8 (for example, the measuring circle shown) they converge to a point lying on this circle. After reflection, these rays also converge to a point lying on this circle.

Actually, though, the points were the rays are deflected are dispersed in the transmission crystal 7 and not all of them lie on the same circle going through the point 8. Although, therefore, the deflected rays are not focussed to a single point, they do form a beam, the sectional area of which at the point 9 in the measuring circle is very small.

Rays having a different wavelength are united in beams which are deflected at different angles and converge to points distributed along the circle circumference. Their intensities may be measured, and then the whole X-ray spectrum may be scanned by displacing a detector 10 along the circumference of the measuring circle.

The angle of incidence of the rays with the lattice planes may be varied by rotating the crystal 7 about a shaft 11 (Fig. 2) lying in the concave surface of the crystal, at right angles to the plane of the drawing and parallel to the lattice planes. The portion of the specimen material surface 1—2 serving as a source of the rays to be measured may then be small, and provision may be made of a directional body 13, for example, a Soller slit collimator, between the surface of the material and the crystal in order to confine the range in which the detector 10 is struck by direct rays. By causing the detector 10 to rotate about the shaft 12 with an angular speed which is twice the angular speed of the crystal 7 about the shaft 11, the detector thus following the cycloidal path designated by the dotted line 14, the detector will occupy, for each position of the crystal 7, exactly that position at which the deflected rays always converge to a substantially point focus. Fig. 3 illustrates one form of apparatus for achieving the desired relative motions. In this embodiment, the detector 10 is secured to an extension 15 of an arm 16 rigidly secured to a shaft 17, on which is seated the curved crystal 7. The extension 15 is pivotably connected to the arm 16, and is adapted to rotate relative to this arm about a shaft 18. The center line of this shaft 18 goes through the center 12 of the measuring circle (Fig. 1). Since the arm 16 always occupies the same position relative to the crystal 7, the shaft 18 describes an arc of a circle when the crystal 7 rotates. At the same time, the extension 15 rotates about the shaft 18 with double the angular speed of the shaft 17 or crystal 7. For this purpose, a toothed wheel or gear 19 is rigidly secured to the extension 15 and centered relative to the shaft 18. The teeth of the wheel 19 engage a toothed rack 20, which is stationary and centered relative to the shaft 17. The diameter of the pitch circle of the toothed wheel 19 is half that of the toothed rack 20.

Upon rotation of the crystal 7 with the aid of a worm 21, driven by an external source of power (not shown), and an associated worm wheel 22, which is seated on the shaft 17, the toothed wheel 19 is displaced along the circumference of the toothed rack 20 and thus performs a rotary motion with the required angular speed.

In Fig. 3, the detector 10, which may be the conventional Geiger-Müller tube, has a cylindrical shape and is provided with a circular inlet window for the rays. In front of the window are provided two diaphragms 23 and 24, which are in turn provided with rectangular apertures. The aperture 25 in the first diaphragm is shaped in the form of a narrow gap, parallel to the rotary shaft 17 of the crystal 7. This gap is located at the area where the beam of rays deflected and focussed by the crystal 7, and emitted by the object specimen surface 1—2, has its smallest sectional area. The aperture 26 of the diaphragm 24 together with the gap 25 constitute a collimator for retaining stray rays.

To insure that the shaft of the detector 10 remains orientated towards the crystal 7, the former is secured, together with the two diaphragms 23 and 24, to a directional arm 27, which is connected rotatably via a stud 28 to the extension 15 of the arm 16. The directional arm 20 is provided with an elongated slot 29, which engages the shaft 17 of the crystal 7. During the displacement of the detector 10 along its scanning cycloidal path, the distance thereof from the crystal varies. This is not prevented by the directional arm 27, since it is adapted to slide along the shaft 17.

The elements of the apparatus determining the position of the detector 10 relative to the crystal 7 must be carefully manufactured, e. g., little backlash, since a small divergence from their correct positions materially affects the accuracy of the resultant measurement. The detrimental effect of inaccurate parts may be reduced by providing a maximum size for the wheel 20 rigidly secured to the support of the detector. It, of course, must be ensured that the wheel centered relative to the crystal shaft should rotate at a suitable rate in opposite sense when the crystal rotates.

While we have described our invention in connection with specific embodiments and applications, other modifications thereof will be readily apparent to those skilled in this art without departing from the spirit and scope of the invention as defined in the appended claims.

What is claimed is:

1. An X-ray fluorescent analysis apparatus comprising a thin curved crystal for deflecting and converging to a point a mono-chromatic beam of X-radiation, said curved crystal having a given radius of curvature and defining a circle of diameter equal to said given radius, detecting means responsive to X-radiation located at said focal point of said X-ray beam to receive said X-radiation, first means for rotating said crystal about an axis extending therethrough at a given angular speed, and second means coupled to said first means for rotating said detecting means about an axis parallel to said crystal and passing through a point substantially at the center of said circle at twice the angular speed of rotation of said crystal.

2. An X-ray fluorescent analysis apparatus as claimed in claim 1, wherein said second means includes a pair of arms pivotable about a common shaft, one of said arms being secured to said crystal, the other of said arms being secured to said detector, said common shaft passing substantially through the center of said circle.

3. An X-ray fluorescent analysis apparatus comprising a thin curved focussing transmission crystal for deflecting and converging to a point, a mono-chromatic beam of X-radiation, a rotary shaft supporting said crystal, said curved crystal being rotatable about said rotary shaft and having a given fixed radius of curvature and defining a measuring circle having a diameter substantially equal to that of said given radius, detecting means responsive to X-radiation located approximately at the focal point of said X-ray beam to receive said X-radiation, an arm having one end rigidly secured to said rotary shaft and rotatable therewith, a stationary first rotary disc aligned with said rotary shaft, an extension pivotably

connected at a pivot point to the other end of said arm and secured to said detector, a second rotary disc rigidly secured to said extension and aligned with said pivot point, and means for rotating said rotary shaft, said first and second rotary discs co-operating to impart a rotation to said detector about said pivot point at an angular speed twice the angular speed of said rotary shaft.

4. An X-ray fluorescent analysis apparatus as claimed in claim 3 in which the first rotary disc is a toothed wheel and the second rotary disc is a toothed rack in engagement with said toothed wheel, the diameter of the pitch circle of said toothed wheel being half that of said toothed rack.

5. An X-ray fluorescent analysis apparatus as claimed in claim 3 in which the detector is rotatably secured to said extension, and a directional arm having a slotted aperture engaging the rotary shaft is secured to said detector to insure that the detector is always properly oriented to said crystal.

6. An X-ray fluorescent analysis apparatus as claimed in claim 3 in which the detector includes a collimating slit located at the focal spot of the X-ray beam and a Geiger-Müller tube for receiving the radiation passing through said slit.

References Cited in the file of this patent

UNITED STATES PATENTS

2,532,810	Harker	Dec. 5, 1950
2,540,821	Harker	Feb. 6, 1951
2,578,722	McCartney et al.	Dec. 18, 1951