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X-RAY SPECTROGRAPH

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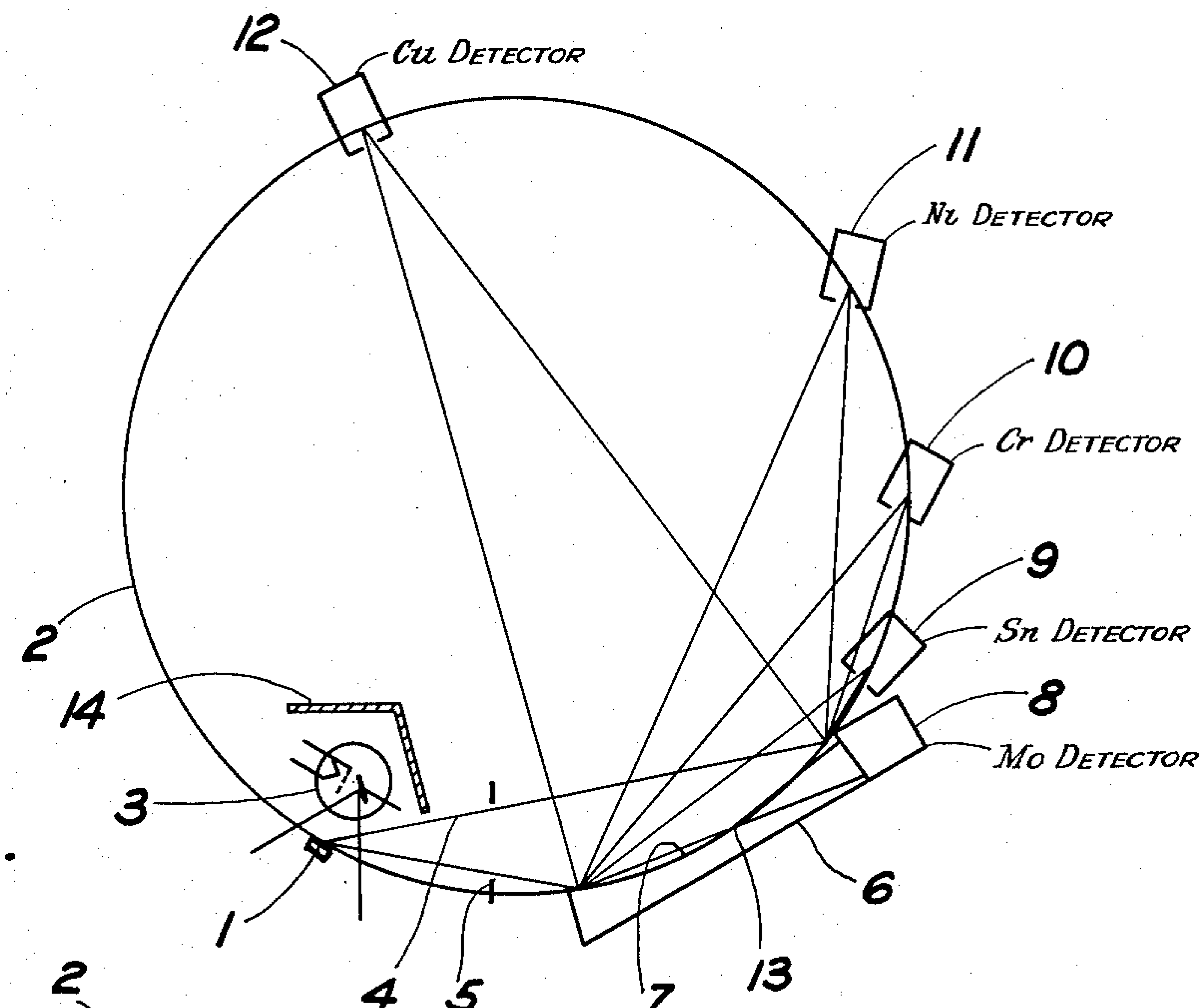

Fig. 1.

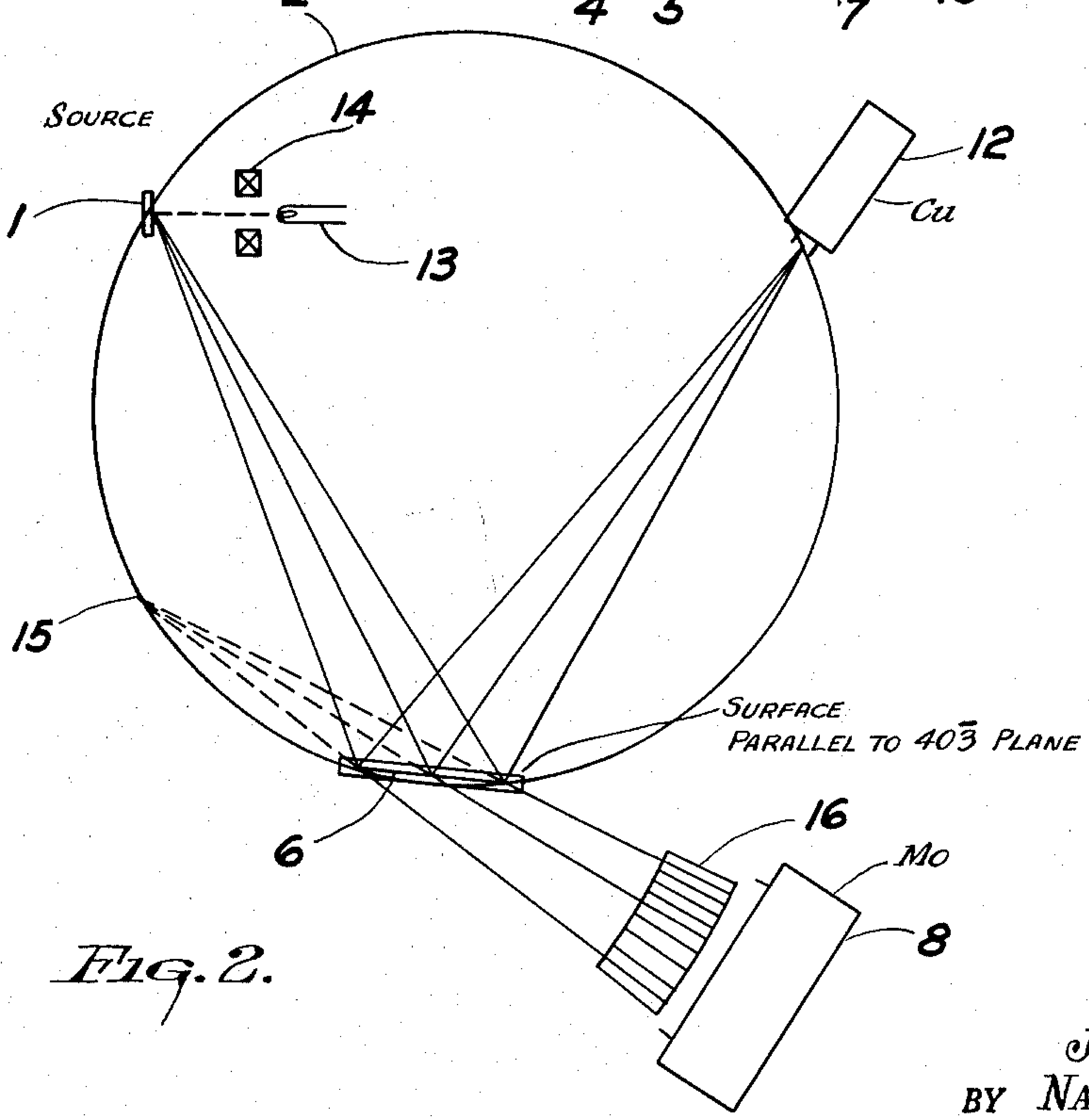

Fig. 2.

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X-RAY SPECTROGRAPH

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Our invention relates to an X-ray spectrograph and more particularly, to an X-ray spectrograph for determining non-destructively, the composition of a material.

In a copending application Serial No. 771,621, filed November 3, 1958, now U.S. Patent 3,046,399, there is described an X-ray spectrograph for determining the constituent elements of the material simultaneously rather than serially. This invention is an improvement in that type of instrument; specifically, the instrument is now adapted for use with a micro-probe X-ray analyzer. More particularly, in the instrument described in that application, a specimen of the material is excited to produce characteristic X-rays of each of the elements in the material. Since each element emits characteristic X-rays which have different wave-lengths, a diffracting crystal which has been cut to diffract radiation of each of a plurality of wave-lengths at angles determined by the reciprocal lattice construction of the crystal is positioned to intercept these characteristic X-rays and reflect the several wave-lengths simultaneously at different angles toward detectors pointed at the diffracting crystal which detect a wave-length corresponding to that of one of the elements.

A principal object of the present invention is to increase the sensitivity of such an instrument.

Another object of the invention is to obtain a higher intensity of the characteristic X-rays from a smaller source.

A further object of our invention is to reduce the background and scattering effects by utilizing receiving slits and detectors of smaller cross-section.

Another object of our invention is to simplify the shielding required with such instruments.

These and further objects of the invention will appear as the specification progresses.

In accordance with our invention we employ a crystal which not only reflects the different wave-lengths corresponding to each of the elements at different angles thereby permitting simultaneous detection of several wave-lengths, but also one which focusses each of the wave-lengths at a series of points conveniently located on a circle, hereinafter referred to as the "focussing circle."

Thus, in accordance with our invention, we employ a crystal which not only satisfies the general conditions specified in the aforesaid copending application, i.e., a crystal which has been prepared to reflect each wave-length at an angle determined by the reciprocal lattice construction of the crystal, but is also deformed into a curved surface so as to converge each of these wave-lengths at a point on the focussing circle. The deformation may either be plastic or elastic or both. For elastic crystals, the crystal is bent and mechanically constrained to maintain the desired curvature. Plastically deformable crystals appropriately shaped may also be used. Consequently, if the detectors are positioned at points on the circle at which the respective wave-lengths are focussed, the intensity of the detected radiation will be greater than if a flat crystal is used which does not focus the radiation.

The crystal we employ is a modified focussing crystal monochromator, i.e., a crystal which has been prepared to reflect each wave-length at an angle determined by the reciprocal lattice construction of the crystal. The reciprocal lattice is a three-dimensional network of points throughout space surrounding each unit cell of the crystal. Each point in the reciprocal lattice is separated from the origin of the reciprocal lattice by a distance inversely proportional to the interplanar spacing of the crystal planes that it represents, and its direction from the origin is exactly the same as the direction of the normal to the planes (cf., X-ray Crystallography, M. J. Buerger, chapter 6, p. 107, et seq., for a more complete discussion of the reciprocal lattice).

The crystal monochromator itself may be of the Johansson-Du Mond type, or of the Johann, Cauchois or logarithmic spiral type. In one embodiment illustrated, we employ a quartz crystal with a surface initially parallel to the $(10\bar{2})$ planes which is then bent to cylindrical shape of radius 2R, with the axis of the cylinder parallel to the *b*-axis of the crystal and the surface ground to a cylinder of radius R (again with axis parallel to *b*). Radiation from a narrow source, i.e., a point or line source, or a source slit placed on the focussing circle (radius R) is diffracted by the crystal and refoccussed, again on the focussing circle, depending upon the wave-length involved, and receiving slits and associated detectors placed at these positions permit simultaneous recording of all wave-lengths.

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

FIG. 1 shows a spectrograph according to the invention employing a reflecting or Johansson-Du Mond type of crystal monochromator; and FIG. 2 shows another embodiment of the invention employing a transmission or Cauchois type of crystal monochromator.

Specimen **1**, located on the circumference of a circle **2** of radius R, which is the radius of the focussing circle, is exposed to a cone of X-rays emanating from a tube **3**, surrounded by shield **14**, emits secondary X-rays whose wave-lengths are characteristic of each of the elements in the specimen. Alternatively, the specimen could be exposed to a focussed beam of electrons of sufficient energy to produce characteristic X-rays from elements in the specimen. The cone **4** of characteristic X-rays from the specimen is limited by a slit **5** and intercepted by a diffracting crystal monochromator **6** whose reflecting surface **7** is ground to a cylinder of radius R so that it forms an arc of the focussing circle. A plurality of detectors **8, 9, 10, 11** and **12** are positioned at various points around the focussing circle and are pointed toward the diffracting crystal to intercept cones of X-rays focussed at those points by a crystal monochromator.

As described in copending application Serial No. 771,621, now U.S. Patent 3,046,399, the crystal is oriented to simultaneously diffract N wave-length into N detectors positioned at various angles with respect to the crystal. The prinicple used to cause simultaneous diffraction into N detectors is governed by satisfying the Laue condition for N characteristic radiations and at least N sets of crystallographic planes (hkl). The simultaneous diffraction from N planes (hkl) corresponding to N characteristic radiations λ_j is accomplished by orienting a specially cut crystal in the following manner.

The three-dimensional reciprocal lattice of the crystal is constructed. N spheres each of radius $1/\lambda_j$ (λ_j is a characteristic wave-length of the jth element, $j=1, 2 \ldots n$), are constructed with a common point of tangency. The common point of tangency of the N spheres is made to coincide with the origin of the reciprocal lattice. With the reciprocal lattice fixed in space, the line of centers of the spheres is rotated about any line drawn through the reciprocal lattice origin. When each and every sphere intersects, or nearly intersects one reciprocal lattice point of a zone in the reciprocal lattice (the zone axis of which is the axis about which the line of centers of spheres is rotated), the condition of simultaneous diffraction for the focussing case is satisfied; all planes (*hkl*) represented by reciprocal lattice points on the circles of reflection are respectively in reflecting positions for the wave-lengths characterized respectively by the spheres (of which the circles are traces); the direction of the line of centers of the spheres is the direction of the incident beam relative to the orientation of the reciprocal lattice. The direction of radii drawn to the reciprocal lattice points lying on the circles define the directions in which the detectors must point relative to the incident beam to receive the diffracted rays.

In this embodiment, crystal **6** has been bent to diffract and reflect the characteristic radiations of Mo, Sn, Cr, Ni, and Cu. If the specimen contains any, or all of these elements, the crystal diffracts and reflects the characteristic radiations of each of the detectors separately. In addition, crystal **6** has been ground to converge the characteristic radiations at points on the focussing circle at which the detectors can be located for receiving a maximum amount of radiation with the smallest receiving slit. An exception has been made in this case for the detector of molybdenum radiation **8** which, because of the exaggerated size of the crystal shown, cannot be located at the focus **13** of the molybdenum characteristic radiation but is positioned to receive a diverging cone of radation.

As in the case described in the copending application, the choice of reciprocal lattice points is not restricted to the zone. For those points not in the zone the focussing properties will be somewhat sacrificed.

In FIG. 2, the specimen, again located on the focussing circle, is exposed to a beam of electrons produced by cathode **13**, and focussed by electron lens system **14**, and emits characteristic X-rays of each of the elements in the specimen. X-rays corresponding to some of the elements are diffracted and transmitted by the crystal **6**, the remainder being reflected and focussed at points on the focussing circle. For those X-rays which are reflected and focussed, the detector is positioned on the focussing circle as described in connection with FIG. 1. In the illustrated embodiment, characteristic X-rays of copper are reflected and focussed at a detector **12** located on the focussing circle.

X-rays of some wave-lengths will not be reflected but will be transmitted through the crystal and will appear to be diverging from a point source on the focussing circle. In this case, the detector is positioned to intercept transmitted X-rays diverging from a "virtual" focus on the focussing circle. Such is the case for molybdenum radiation for which detector **8** is positioned behind the crystal and intercepts diffracted X-rays appearing to diverge from the virtual focus **15**. To detect substantially only the X-rays diverging from this virtual focus, a converging Soller slit assembly **16** is interposed between the detector and the crystal. Also, in this case, a large aperture detector is employed.

While we have thus described our invention with a specific embodiment, other modifications will be apparent without departing from the spirit and scope of the invention as defined in the appended claims.

What we claim is:

1. An X-ray spectrograph for determining the constituent element of a material comprising means to excite a specimen of the material to produce characteristic X-rays, a plurality of detectors, and a stationary focussing diffracting crystal positioned to intercept and diffract the characteristic X-rays emanating from said specimen, said crystal being deformed to diffract and focus radiation of each of a plurality of wave-lengths to one of a plurality of reciprocal lattice points of the crystal, and a plurality of detectors each of which is located at a reciprocal lattice point on the focussing circle of said crystal.

2. An X-ray spectrograph for determining the constituent elements of a material comprising means to excite a specimen of the material to produce characteristic X-rays, a plurality of detectors, and a stationary focussing diffracting crystal positioned to intercept and reflect characteristic X-rays of one wave-length and to transmit characteristic X-rays of another wave-length, said crystal being deformed to diffract and focus each of said wave lengths to one of a plurality of reciprocal lattice points of the crystal, and a plurality of detectors each of which is located at a reciprocal lattice point on the focussing circle of said crystal.

3. An X-ray spectrograph for determining the constituent elements of a material comprising means to excite a specimen of the material to produce characteristic X-rays, a plurality of detectors, and a stationary focussing diffracting crystal positioned to intercept and reflect characteristic X-rays of a plurality of wave-lengths, said crystal being deformed to reflect and focus X-rays of each of said wave-lengths to one of a plurality of reciprocal lattice points of the crystal, and a plurality of detectors each of which is located at a reciprocal lattice point on the focussing circle of said crystal.

4. An X-ray spectorgraph for determining the constituent elements of a material comprising means to excite a specimen of the material to produce characteristic X-rays, a plurality of detectors, and a stationary focussing diffracting crystal positioned to intercept and transmit characteristic X-rays of a plurality of wave-lengths, said crystal being deformed to transmit and focus X-rays of each of said wave-lengths to one of a plurality of reciprocal lattice points of the crystal, and a plurality of detectors each of which is located at a reciprocal lattice point on the focussing circle of said crystal.

5. An X-ray spectrograph for determining the constituent elements of a material comprising a source of X-rays, means to expose a specimen of said material to said X-rays to produce fluorescent characteristic X-rays therefrom, a plurality of detectors, and a stationary focussing diffracting crystal positioned to intercept and diffract the characteristic X-rays from said specimen, said crystal being deformed to diffract and focus X-radiation of each of a plurality of wave-lengths to one of a plurality of reciprocal lattice points of the crystal, and a plurality of detectors each of which is located at a reciprocal lattice point on the focussing circle of said crystal.

6. An X-ray spectrograph for determining the constituent elements of a material comprising a source of electrons, means to expose a specimen of said material to said electrons to produce characteristic X-rays therefrom, a plurality of detectors, and a stationary focussing diffracting crystal positioned to intercept and diffract the characteristic X-rays from said specimen, said crystal being deformed to diffract and focus X-radiation of each of a plurality of wave-lengths to one of a plurality of reciprocal lattice points of the crystal, and a plurality of detectors each of which is located at a reciprocal lattice point on the focussing circle of said crystal.

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