July 15, 1958 J. HILLIER 2,843,750

X-RAY SPECTROMETER

Filed Dec. 7, 1954

Fig. 1.

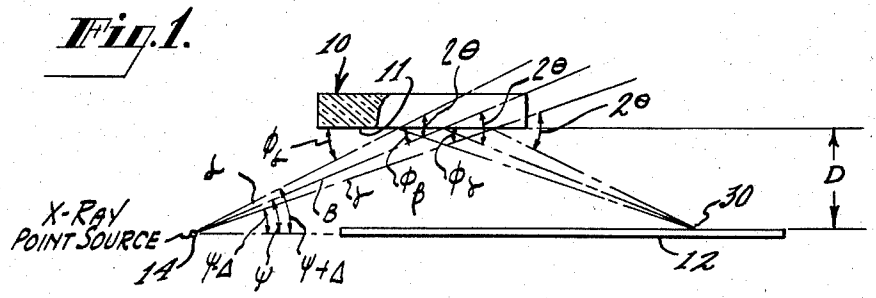


Fig. 2.

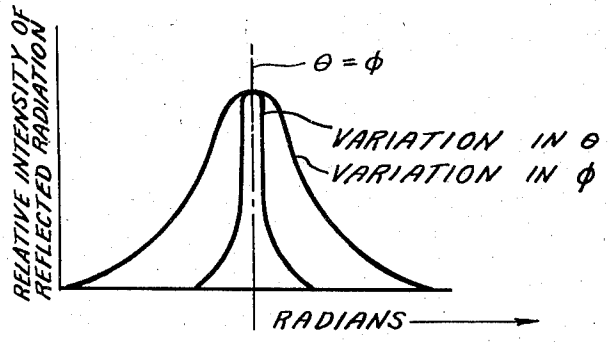


Fig. 3.

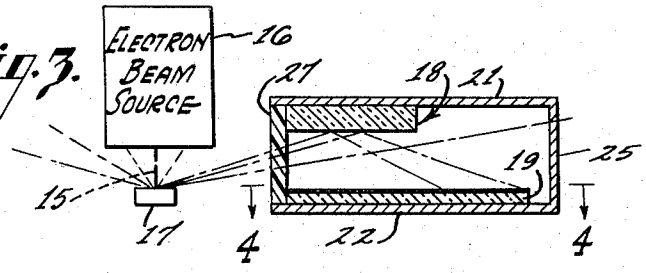
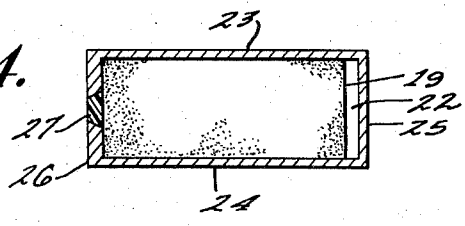


Fig. 4.



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X-RAY SPECTROMETER

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10 Claims. (Cl. 250—53)

This invention relates to a new and improved X-ray spectrometer that is particularly useful in microanalysis. An X-ray spectrometer, according to the present invention, provides a well defined spectrum from X-rays generated in a minute cross-sectional area of an object under observation.

Means are presently available for irradiating a minute area of a material with high velocity electrons, such means embody the techniques of obtaining a sharply defined electron probe. It is known that X-ray emission occurs from the electron irradiated area of the material under test. If a fine electron probe is used to irradiate the material, X-ray emission then takes place from what is substantially a point source. The X-ray emission from this point source occurs in all directions.

It is an object of this invention to provide a new and improved X-ray spectrometer for the analysis of X-rays emitted from substantially point sources of X-rays.

It is another object of the present invention to provide an X-ray spectrometer which can be brought in close proximity to the source of X-rays and which thereby can produce high intensity spectra.

It is a further object of the present invention to provide an X-ray spectrometer requiring no moving parts.

It is a still further object of the present invention to provide an X-ray spectrometer which incorporates focusing action for X-rays having the same wavelength.

These and other objects of the present invention are provided by an X-ray spectrometer constructed according to one embodiment of the present invention. This X-ray spectrometer comprises a flat crystal having lattice planes parallel to, and at some distance from, a sensitive X-ray detector such as a photographic emulsion. The crystal and photographic plate may be placed in a container which excludes all other types of radiation to which the photographic emulsion is sensitive. The X-ray source is arranged in the plane of the photographic emulsion. Radiation is reflected from the crystal according to the existence of Bragg law conditions. Thus, X-rays having different wavelengths are reflected to different points on the photographic emulsion to provide a spectrum. This spectrometer may be very small so that it is possible to locate the spectrometer very close to the source of X-rays. The intensity of the X-rays striking the photographic emulsion will be increased accordingly. A further increase in intensity is obtained because of the geometry of the device.

Still further objects and advantages of this invention will, of course, become apparent and immediately suggest themselves to those skilled in the art to which the invention is directed from a reading of the following specification in connection with the accompanying drawings in which:

Figure 1 is a ray diagram of apparatus for explaining principles of operation thereof;

Figure 2 is a graph of the relative intensity of the reflected radiation from the crystal according to the angular orientation of the crystal with respect to incoming and reflected X-rays;

Figure 3 is a sectional view of X-ray spectrometer constructed according to one embodiment of the present invention; and

Figure 4 is a sectional view of Figure 3 taken along the section line 4—4 as viewed in the direction of the arrows.

Referring to Figure 1, a sectional view through a crystal **10** is shown. A flat surface **11** forms the active face of this crystal. This crystal may be grown or cut so that the atoms in a major portion of the crystal structure lie in lattice planes which are parallel to the active face of the crystal. The surface **11** is desirably planar and is the largest surface of the crystal **10**. The crystal may, therefore, be in the form of a relatively flat sheet. In the exemplary crystal shown in Figure 1, the selected largest surface **11** appears at the lower side of the crystal **10**. For analytical applications the interplanar spacing of the atomic planes making up the structure of the crystal is desirably large. A large interplanar spacing permits the production of a spectrum covering a wide range of wavelengths which may include the lower wavelengths which are useful in many applications.

A photographic plate **12** is located below the crystal **10**. This photographic plate **12** is coated with a photographic emulsion which is sensitive to X-rays. It is desirable for the photographic plate **12** to have a planar surface. The planar surface of the photographic plate **12** is arranged to lie parallel to the selected crystal face **11** on the lower side of the crystal **10**. It may be observed at this time that other X-ray responsive surfaces which are preferably planar may be alternatively used instead of the photographic plate **12**. The X-rays to be analyzed are generated in the point source **14** which is located in the plane of the photographic plate **12**. Generation of a point source of X-rays will be more fully discussed in connection with the embodiment of the present invention shown in Figure 3. However, known methods of generating a substantially point source of X-ray radiation may be employed to provide the point source of X-rays **14**.

In the operation of Bragg law spectrometers it is generally necessary for a great many angles of incidence with the crystal to be provided for all wavelengths of the X-ray radiation to be analyzed. This requirement is generally imposed so that Bragg law conditions occur for the selection of the desired wavelengths to provide a spectrum of the X-ray radiation. The spectrum may be detected and recorded on charts or a photographic plate, for example. However, the intensity and general readability of the spectrum may leave much to be desired. With the use of the spectrometer provided by the present invention for analyzing radiation from a point source of X-rays, each X-ray wavelength line present will be made sharp and intense to provide more readable spectra. The geometrical arrangement of the spectrometer proposed herein provides a focussing action for each and all wavelengths present by overcoming many of the limitations imposed by previously known spectrometers.

Three rays α, β and γ of the same wavelength are shown emanating from the point source of X-rays **14**. The focusing action of the present invention will be illustrated herein for rays of the same wavelength. It will be apparent that a similar focusing action will be obtained for rays of different wavelength in the production of an X-ray spectrum.

The incident ray α is emitted from the point source **14** at an angle $\psi+\Delta$. The next ray β is emitted from the point source **14** at an angle ψ with respect to the plane of the photographic plate **12**. The other incident ray γ is emitted from the point source at an angle of $\psi-\Delta$ with respect to the plane of the photographic plate. The rays α, β and γ are therefore, incident upon the crystal face at angles ϕ_α, ϕ_β and ϕ_γ respectively. The angle $\phi_\alpha=\psi+\Delta$; the angle $\phi_\beta=\psi$; and the angle

$\phi_\gamma = \psi - \Delta$. It is generally known that incident X-rays will interact within the atomic structure of a crystal and will be reflected from the crystal when the Bragg condition for the crystal and the radiation is satisfied. This condition is defined in the Bragg equation,

$$n\lambda = 2d \sin \theta$$

where n is an integer corresponding to the order of the reflection, λ is the wave length of the given radiation, d is the interplanar spacing between the atomic planes of the crystal, and θ is one half the angle of total deviation between the transmitted and reflected rays.

It has been found that reflection from the crystal takes place although the angle of incidence ϕ is not exactly equal to the angle θ stated in the Bragg equation. However, it is known that the total deviation of all of the reflected radiation is always very close to twice the Bragg angle θ as defined in the above-stated equation. These two realizations together with certain geometrical relationships will lead to an understanding of the operation of the spectrometer proposed by the present invention and of its focusing action.

In Figure 2 the relationships between the intensity of reflected radiation from the crystal 10, the angle of incidence ϕ, and one half of the angle of total deviation θ are plotted. For the large crystals, which are preferably used in this embodiment of the present invention, the intensity of the reflected radiation is critically dependent on the value of the angle of total deviation which is expressed as its half angle θ, and falls off sharply for values of the angle θ which depart from the value of θ which satisfies the above-stated Bragg equation. On the other hand, the intensity of the reflected radiation is not so critically dependent on the angle of incidence ϕ. The intensity of reflected radiation falls off more slowly as the angle of incidence ϕ departs from values that equal half the angle of total deviation θ which satisfies the Bragg equation. This result is known as the "rocking curve" phenomenon, and can be derived from the theory of X-ray diffraction.

Returning to Figure 1, it is assumed that the angle of incidence ϕ_β of the ray β on the surface **11** of the crystal **10** is equal to one half the angle of total deviation θ which satisfies the Bragg equation. This ray β is, therefore, reflected and intercepts the photographic plate **12** at a point 30 along the spectral line for all X-rays of this same wavelength. The geometry of the situation will determine the lateral position of the point **30** on the photographic plate **12**. Application of the Bragg equation to the geometry shows that the distance from the point source of X-rays to the focus point is equal to,

$$\frac{4Dd}{n\lambda}\sqrt{1-\left(\frac{n\lambda}{2d}\right)^2}$$

where D is the distance between the plane of the photographic plate **12** and the crystal face **11** and the other parameters have been previously defined in connection with the Bragg equation.

Although the angles of incidence ϕ_α and ϕ_γ of the rays α and γ upon the crystal surface **11** are slightly more and slightly less than the angle ϕ_β respectively, they are reflected. The relationship plotted in Figure 2 shows that this is possible. This relationship also shows that the total deviation of the reflected radiation is very nearly equal to the angle 2θ. Consequently the reflected rays α and γ intercept the photographic plate **12** at substantially the same point **30** as the other ray β. It is, therefore, apparent that a measurable focusing action is obtained whereby the intensity of the spectral line is enhanced.

Referring to Figure 3 a sectional view of an embodiment of a spectrometer according to the present invention is shown in conjunction with a source of X-rays produced by the irradiation of a minute cross-sectional area of a

surface of a material under observation by a fine beam or probe **15** of fast moving electrons. An electron beam source **16** may comprise any known electron accelerating device. For example, an electron gun for producing a beam of electrons and an electron lens system for forming a fine probe may be used. It should be understood, however, that any other means for producing an electron probe having the desired characteristics may be employed. An object **17** under observation is preferably in the form of a thin section. High velocity electrons from the electron beam source **16** striking the object interact with the atomic srtucture thereof, and produce a point source of X-rays. It is preferable that the electron beam source and object are enclosed in a vacuum chamber. However, the spectrometer may be located internally or externally as desired for a particular design.

The crystal **18** and the photographic plate **19** which are used in this embodiment of an X-ray spectrometer are enclosed in a container **20**. The container **20** is a rectangular box having four sides **21**, **22**, **23** and **24**, and two end walls **25** and **26**. Figure 4 presents another view of the container. The four sides **21**, **22**, **23** and **24**, and the end wall **25** on the right are preferably constructed of a non-crystalline material having a low atomic weight. The use of a material having a low atomic weight reduces the scattering of X-rays from the walls of the container since X-rays are not readily reflected from such substances and may pass through them more easily. The opposite end wall **26**, located on the left side of the container **20**, is preferably constructed of a material having a high atomic weight. Lead, for example, may be used. A slot or opening **27** is provided in this lead wall **26**. The opening **27** normally is located in the center of the wall and extends between the top and the bottom of the container. The opening **27** may be covered with a very thin layer of light-opaque and electron-opaque but X-ray transparent material. This covering material is preferably noncrystalline. For example, carbon-black filled plastic may be found useful. The point source of X-rays is so located that the X-ray source lies on a line which is in the plane of the photographic plate **19**. It is desirable that this line bisect the container **20**. In this manner X-rays penetrate the material in the opening or slot **27** on the left hand end wall **26** and produce a spectrum on the photographic plate **19** after reflection from the crystal **18**. The crystal **18** is mounted on the upper wall **21** of the container **20**. The sensitive photographic emulsion **19** is located on the bottom wall **22**. As was previously mentioned, the crystal face is parallel to the photographic plate by suitable mounting.

The emulsion on the photographic plate **19** is preferably selected for highest sensitivity to X-ray radiation for the wave-length expected. The dispersion of the X-ray spectrum is determined in part by the distance between the crystal face and the photographic plate. The length of the container end of the photographic plate is determined by the spectrum of the radiation. It may be determined for the particular conditions expected upon consideration of the above-stated equation which locates the distance between a focus point and the source of X-rays. Solely by way of example, the dimensions of the container an X-ray spectrometer, which may be provided according to the present invention, is two inches in length, one inch in width and one-quarter inch in height. The noncrystalline material of which the side walls **21** to **24** and the end wall **25** of the container **20** are constructed may be plastic.

What is claimed is:

1. An X-ray spectrometer comprising a crystal having a selected set of crystal lattice planes, an X-ray sensitive device having a planar surface, means for locating said surface in a plane parallel to said selected set of crystal lattice planes, and means for positioning a substantially point of source of X-rays to be analyzed in the plane of said surface so that radiations from said source will im-

pinge upon said crystal and will be diffracted therefrom onto said surface.

2. An X-ray spectrometer for analyzing radiation from a point source of X-rays comprising a container having at least two parallel sides, a crystal attached to one of said sides, and a device having an X-ray responsive surface, said device being mounted on the other of said parallel sides with its said surface opposed to said crystal, said crystal having a face and lattice planes disposed parallel to said surface, and said container being positioned in proximity to said point source with said point source in the plane of said surface so that radiations from said source will impinge upon said crystal and will be diffracted therefrom onto said surface.

3. In an X-ray spectrometer apparatus for analyzing the atomic composition of an object under observation wherein a predetermined cross-sectional area of said object is electron irradiated to provide a substantially point source of X-rays to be analyzed, and X-ray spectrometer comprising an X-ray responsive element located in the plane of said cross-sectional area, a crystal with substantially parallel planes of atoms forming the atomic structure thereof, said planes of atoms being located adjacent and parallel to a surface of said crystal, and said crystal being supported near said X-ray responsive element with said surface parallel and opposite to said X-ray responsive element so that radiations from said source will impinge upon said crystal and will be diffracted therefrom onto said element.

4. A spectrometer for providing X-ray spectra comprising an X-ray detector element having a planar X-ray responsive surface, a crystal, and means for maintaining said crystal and said X-ray detector element in such positions that lattice planes and a face of said crystal and said X-ray responsive surface are opposite and parallel to each other whereby radiations from a substantially point source of X-rays disposed in the plane of said surface will impinge upon said crystal and will be diffracted therefrom onto said surface.

5. A spectrometer for providing X-ray spectra comprising a photographic emulsion, a crystal having a set of crystal lattice planes and a face, a container, said photographic emulsion being held in a first position by said container, and said crystal being held in a second position by said container such that said lattice planes and said face are opposite and parallel to said photographic emulsion whereby radiations from a point source of X-rays disposed in the plane of said photographic emulsion will impinge upon said crystal and will be diffracted therefrom onto said photographic emulsion.

6. A spectrometer for providing X-ray spectra from a point source of X-rays comprising a photographic emulsion, a crystal having a planer crystal face, said crystal having a selected set of crystal lattice planes parallel to said face, a holder, said photographic emulsion being held in a first position that is coplanar with said point source of X-rays by said holder, said crystal being held in a second position with its face parallel and opposite to said photographic emulsion by said holder and means to dispose said point source of X-rays in the plane of said photographic emulsion so that radiations from said source will impinge upon said crystal and will be diffracted therefrom onto said photographic emulsion.

7. Spectrometric apparatus for analyzing radiation emanating from substantially a point source thereof comprising a detector crystal having a planar surface parallel to a selected set of lattice planes therein, and a photographic plate spaced from said crystal and parallel to the planar surface thereof, said point source of radiation being positioned in the plane of said photographic plate so that radiations from said source will impinge upon said crystal and will be diffracted therefrom onto said photographic plate.

8. An X-ray spectrometer for analyzing X-rays emanating from a point source on a material under study comprising a rectangular container having opposite sides thereof disposed in parallel planes, a crystal having a desired set of crystalline planes located parallel to a surface thereof, said crystal being mounted on one of said opposite sides of said container, a photographic emulsion, said photographic emulsion being arranged on another of said opposite sides parallel to said crystalline planes, and means for providing in the plane of said photographic emulsion said point source of X-rays from said material whereby radiations therefrom will impinge upon said crystal and will be diffracted from said crystal onto said photographic emulsion.

9. In an X-ray spectrometer apparatus wherein a substantially point source of X-ray is provided, the combination comprising a photosensitive surface that is responsive to X-ray radiation, said surface being mounted in the plane of said point source, and a crystalline substance having parallel crystal lattice planes parallel to a face of said crystalline substance, said crystalline substance being mounted in a position so that said face is spaced from and parallel to said surface whereby radiations from said source will impinge upon said crystalline substance and will be diffracted therefrom onto said surface.

10. In an X-ray spectrometer apparatus for analyzing the atomic composition of a material including means for electron irradiating a minute cross-sectional area of said material whereby X-ray emission from said area of said material occurs, a spectrometer comprising an enclosure, said enclosure having side walls and two end walls, said side walls being constructed of a material characterized by a low atomic weight, one of said end walls of said container being composed of an X-ray absorbing material having an opening therein, said opening being filled with a noncrystalline, substantially x-ray transparent material, said other end wall of said container being composed of material having a low atomic weight, said first-named end wall of said enclosure being located near said minute area of said material whereby X-rays will penetrate through said opening, a photographic emulsion, said photographic emulsion being affixed on one of said side walls of said enclosure, said enclosure being located with said photographic emulsion in the plane of said minute area of said material, and a crystal having crystal lattice planes therein parallel to a surface thereof, said crystal being attached to another one of said side walls of said container which is opposite to said side wall of said container to which said photographic emulsion is affixed, and with said surface of said crystal located opposite and parallel to said photograph emulsion.

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