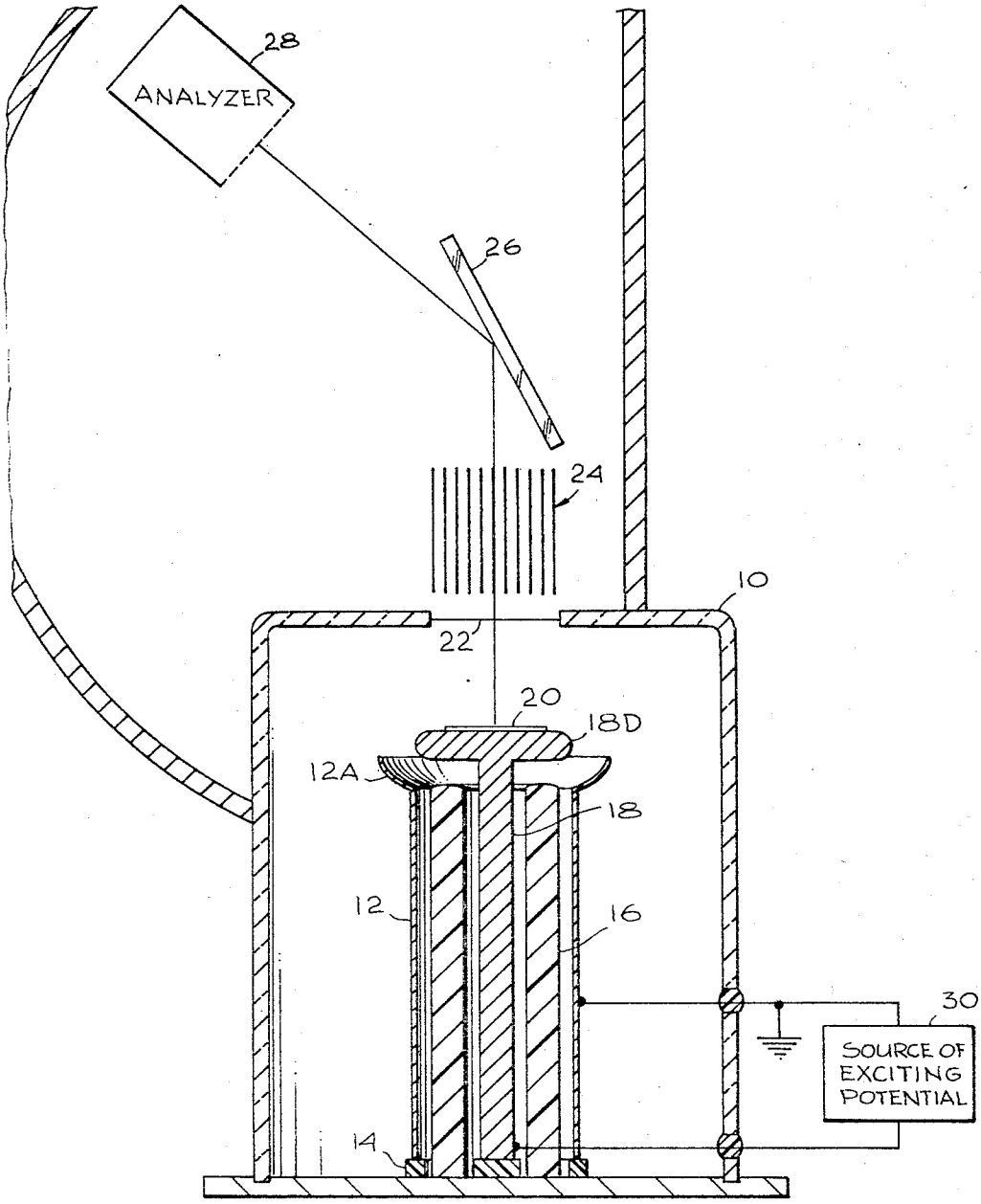
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GASEOUS GLOW DISCHARGE TUBE WITH CATHODE MEANS SURROUNDING ANODE MEANS

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GASEOUS GLOW DISCHARGE TUBE WITH CATHODE MEANS SURROUNDING ANODE MEANS

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U.S. Cl. 250—51.5 7 Claims

ABSTRACT OF THE DISCLOSURE

A sample to be analyzed is placed on the anode of a cold cathode gaseous glow discharge tube. The cathode effectively surrounds the anode and bombards the sample with electrons whereby X-ray fluorescence is excited, which is then used for analysis.

This invention relates to apparatus used for the analysis of materials wherein X-ray fluorescence is induced by the incidence of an electron beam on the material being analyzed, and more particularly to improvements therein.

The presently employed techniques for achieving X-ray fluorescence of a material which is being studied, is to derive the bombarding electron beams from an electron gun in a cathode ray tube, which usually gives up to 50 kev. electrons. A thin exit window is employed at one end of this tube for enabling the beams to exit into the air. The equipment usually requires power supply regulators and other apparatus necessary to operate the electron gun.

An object of this invention is to provide a simpler source for generating electrons for bombarding materials to achieve X-ray fluorescence of such materials.

Another object of this invention is the provision of a novel and useful arrangement for securing bombarding electrons for X-ray fluorescent analysis.

Still another object of the present invention is the provision of a relatively inexpensive system for generating electrons for bombarding a specimen to achieve X-ray fluorescence thereof.

These and other objects of the present invention are achieved by placing the sample to be analyzed on the anode of a cold cathode gaseous glow discharge arrangement. If the sample is an insulator, it may be coated with a metallic conducting layer (much thinner than the electron energy range) in order to prevent charge accumulation on the sample surface. The bombardment by energetic electrons from the gas discharge will excite X-ray fluorescence of the constituents of the sample, providing that the high voltage across the discharge is made greater than the binding energies of either K, L, or M electron shells of the sample constituents. The spectrum of fluorescent X-rays thus excited may be examined by allowing a portion of these X-rays to pass a thin window adjacent to the discharge into an X-ray spectrometer for analyzing the spectrum.

The novel features that are considered characteristic of this invention are set forth with particularity in the appended claims. The invention itself both as to its organization and method of operation, as well as additional objects and advantages thereof, will best be understood from the following description when read in connection with the accompanying drawing, which is a schematic diagram of an embodiment of the invention.

Referring now to the drawing, which is a schematic illustration of the invention, walls **10** enclose an evacuated space in which a suitable gas for supporting a glow discharge is employed. The gas may be argon, neon, or xenon,

or other such inert gases. Within the space enclosed by the walls **10** there is placed a cathode structure **12** which, by way of illustration, has the form of a cylinder with an outward flaring end **12A**. The cathode **12** is supported on an insulating ring **14**. Within the cylinder **12**, there is placed a second shielding cylinder **16** made of insulating materials. An anode **18**, comprising a long narrow rod which extends along the axis of the cylinder **12**, has a disc **18D** supported at right angles to the rod and attached to the end thereof which extends adjacent to the outward flanged end **12A** of the cathode **12**. A specimen **20** which is to be analyzed is placed on the disc portion of the anode.

A thin window **22**, which can be penetrated by X-rays emitted from the material **20**, is placed in the walls **10** adjacent the specimen **20**. An X-ray collimator **24** is placed adjacent the thin window. X-rays from the specimen can pass through the thin window and the collimator to strike the analyzing crystal of the X-ray spectrometer, which is placed at the Bragg angle to reflect these X-rays into the detector **28**.

The cathode **12** is connected to ground and to a source of exciting potential **30**, and the anode is also connected to this source of potential. Connections are made through insulated openings in the walls **10**. While a cold cathode discharge may be established in the gas within the envelope **10** by connecting the anode and cathode to a source of high current, high voltage DC, it is preferred to use a source of radio frequency energy to induce the glow discharge. With DC glow discharge excitation, it is necessary to coat the surface of a sample which is an insulator with a thin conductive film to eliminate any charge collection. The thickness of this film should be insufficient to prevent electron penetration. This is not necessary with RF excitation.

The electrons liberated by the glow discharge established between the anode and the cathode bombard the sample **20** whereby X-ray fluorescence of the constituents of the sample are excited, providing that the high voltage existing across the discharge is somewhat greater than the binding energies of either K, L, or M electron shells in the sample constituents. A spectrum of fluorescent X-rays thus excited is examined when the X-rays pass through the thin window which is positioned adjacent to the discharge into the X-ray spectrometer comprising the collimator, crystal, and detector.

The size of the electron current attainable to bombard a sample can easily be several thousand times as large as the bombarding current which is easily obtained from an electron gun. Such an increase in the intensity of X-rays thus excited can provide a higher resolution of the spectrum and greater sensitivity to measuring small amounts of contamination in samples.

Because no difficulties are present of the type due to low electron energy in an electron gun, lower energy electrons can be used to bombard the sample than would be practical from an electron gun. As a consequence of this, shallower layers in a sample may be studied whereby the depth resolution of analysis may be improved.

By using the radio frequency source of exciting potential, instead of a direct current source of exciting potential, it has been indicated that an insulator may be analyzed without the need for a charge draining metallic coating. In this instance, the X-rays are observed which are produced during the half cycle when the sample is bombarded by the electrons. During the other or negative half cycle of voltage, the sample will be ion bombarded and thus its surface layer may be sputtered away allowing a fresh layer, previously deep in the sample, to be analyzed, and ultimately yielding an analysis-versus-depth for the sample.

There has accordingly been described herein a novel,

useful and unique arrangement for bombarding a sample with electrons having an energy sufficient to cause X-ray fluorescence of samples. While a specific embodiment of the electron producing structure has been described and claimed, it will be appreciated that those skilled in the art may depart from the structure shown without departing from the spirit of this invention nor the intended scope of the claims.

What is claimed is:

1. Apparatus for causing X-ray fluorescence of the surface of a sample comprising walls defining an enclosed chamber, an inert gas within said chamber, means for supporting said sample within said chamber, electrode means for establishing a gaseous glow discharge within said chamber adjacent said sample for bombarding said sample with electrons produced by said gaseous glow discharge, and thin window means in the wall of said chamber for passing X-rays produced by said electron bombardment of said sample out from said chamber.

2. Apparatus as recited in claim 1 wherein said means for establishing a gaseous glow discharge comprises an anode, a cathode spaced from said anode, said specimen being placed upon said anode, and means for applying a potential to said anode and cathode for establishing said glow discharge.

3. Apparatus as recited in claim 2 wherein said cathode has the shape of a hollow cylinder having an outward flare at one end, and said anode has the shape of a rod terminating in a disc positioned at right angles to said rod, said rod being concentric with said hollow cylinder and said disc being adjacent the outward flare of said cathode.

4. Apparatus as recited in claim 2 wherein said means for applying a potential to said anode and cathode applies said potential at a radio frequency.

5. Apparatus for bombarding the surface of a specimen with electrons to produce X-ray fluorescence comprising an evacuated envelope filled with an inert gas, an anode, a cathode spaced from and surrounding said anode, said anode having a surface adapted for supporting said specimen, means for applying a potential between said anode and cathode for establishing a glow discharge therebetween and for bombarding said specimen with electrons, a thin film window in the walls of said chamber adjacent said anode for passing therethrough X-rays emitted by said specimen in response to said bombardment, and X-ray reflection means positioned adjacent said thin film window for measuring the X-rays passing therethrough.

6. Apparatus as recited in claim 5 wherein said means for establishing a potential across said anode and cathode comprises a source of radio frequency potential.

7. Apparatus as recited in claim 5 wherein said cathode has the shape of a hollow cylinder having an outward flare at one end, and said anode has the shape of a rod terminating in a disc positioned at right angles to said rod, said rod being concentric with said hollow cylinder and said disc being adjacent the outward flare of sad cathode.

References Cited

UNITED STATES PATENTS

3,235,727 2/1966 Shapiro.

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313—55, 59