

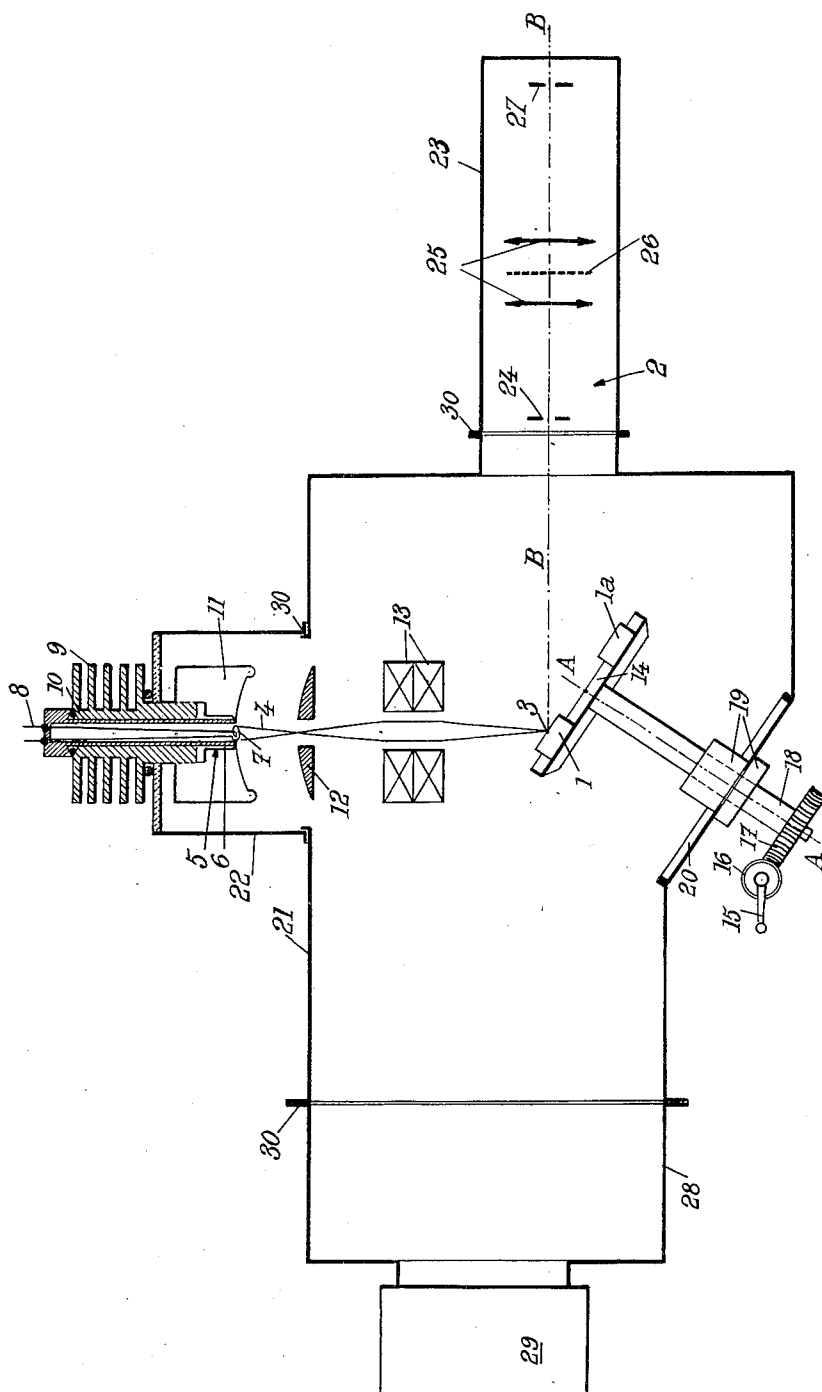
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METHODS AND DEVICES FOR PERFORMING SPECTRUM ANALYSIS, IN
PARTICULAR IN THE FAR ULTRAVIOLET REGION

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METHODS AND DEVICES FOR PERFORMING SPECTRUM ANALYSIS, IN PARTICULAR IN THE FAR ULTRAVIOLET REGION

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The present invention relates to methods and devices for performing spectrum analysis, in particular in the far ultraviolet region, that is to say concerning elements of low atomic number. This invention is more especially, but not exclusively, concerned with the analysis of alloys, and in particular steels.

The chief object of this invention is to provide a method and device of the above mentioned kind which are better adapted to meet the requirements of practice than those used up to this time, especially concerning the purity of the spectrum lines and the positioning, brightness and stability of the emission source.

According to the present invention, to perform spectrum analysis of a sample placed in a vacuum together with optical means for dispersing the radiation emitted from said sample, the emission of characteristic spectrum lines from a given zone of the sample is obtained by bombarding such a zone, of extremely small area, with an intensive electron beam focussed on said zone, whereby this zone is heated to very high temperature and emits spectrum lines in the range of visible and ultraviolet rays.

The invention is more particularly concerned with the application thereof to spectrometers for the analysis of elements of low atomic number, such as carbon, sulphur, phosphorus, oxygen and nitrogen in steels.

The only FIGURE of the drawing diagrammatically shows, in section, the emission chamber of a spectrometer made according to the present invention.

The sample 1 to be analyzed and the optical dispersion system 2 are placed in a vacuum chamber and the emission of the characteristic spectrum lines of a given zone 3, of extremely small area, of said sample 1 is obtained by bombarding this zone by means of an intensive electron beam 4 produced by an electron gun 5 and focussed on said zone, which is thus brought to a very high temperature and emits spectrum lines in the range of visible or ultraviolet rays.

The electron gun 5, of a type known in itself, includes, in the example shown:

(a) An electrode carrier 6 with a filament or cathode 7 heated and carried by conductors 8 at a high negative voltage, generally higher than 1500 volts, so as to supply an intensive electron beam 4; evacuation of the heat produced at the cathode is obtained by means of the cooling fins 9 of a piece 10 made of a material which is a good conductor of heat, said piece 10 surrounding the filament;

(b) A control electrode 11, of the Wehnelt or Pierce type, generally at the same potential as the filament, and an anode 12 which is grounded, these two last mentioned electrodes having suitable shapes to achieve a satisfactory focussing of the electron beam 4 with a high density of electrons, corresponding for instance to a current of 20 ma. under a voltage of 20 kv.; and

(c) Magnetic or electrostatic lenses 13 which make it possible to act upon beam 4 so as to vary the diameter of the spot (which may be reduced for instance to an area of 0.01 sq. mm., which permits of applying an intensity of 4 mw./sq. cm. in zone 3) and to displace the impact zone 3 of the electrons upon sample 1.

The various samples 1, 1a are carried by a kind of magazine including a disc 14 which is rotatable about an

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axis AA (for instance by means of a crank handle 15 which drives, through worm and worm wheel means 16, 17, the shaft 18 on which said disc 14 is mounted). This shaft 18 passes through gas-tight joints 19 disposed on either side of a plate 20, which is removably mounted, for instance by screwing, in the wall of the vacuum chamber. With such an arrangement, it is possible to bring at will any of the samples, such as 1, 1a, and even any desired zone thereof, at the impact point of the electrons without having to open the vacuum chamber.

The vacuum chamber includes, in the example shown, four portions, to wit:

(1) A portion 21 in which is disposed the sample carrier and the lenses 13;

(2) A high voltage chamber portion 22 in communication with said portion 21 and in which is mounted the high voltage system of the electron gun;

(3) A portion 23 which contains the optical system 2 having its axis at BB and including, in the known manner, an entrance slit 24, lenses 25, a dispersion element such as a diffraction grating 26 or a prism, and an exit slit 27;

(4) An evacuation portion 28 in which a vacuum, advantageously averaging 10^{-4} mm. of mercury, is obtained by a system of pumps 29.

Flanges 30 permit of connecting the various portions 21, 22, 23, 28 together while keeping them under the vacuum produced by pump 29.

The operation of the spectrometer which has been described is as follows.

The impact of the electrons produced by gun 5 and focussed on the zone 3 of sample 1 causes a melting, and possibly a volatilization, of the components of this zone. Due to the fact that an electron gun of the space charge type as illustrated permits of obtaining a focussed electron beam having a high specific intensity, zone 3 is excited so as to become a spectroscopic source in the range of visible and ultraviolet rays, ensuring a brightness at least equal to that of the electric arc. As, on the other hand, a vacuum is ensured, it is possible to excite light elements, such as carbon, sulphur, phosphorus, and gases, such as oxygen, nitrogen, halogens (both in the dissolved form and in the form of chemical compounds), which is very advantageous for the analysis of steels and other alloys.

When it is desired to perform an analysis point by point, it is advantageous to feed the cathode of the electron gun with very short pulses, for instance of a duration of some alternations, so as to obtain a very high temperature local heating of a given point of the sample, without enabling the thermal conductivity to produce a thermal excitation of the adjoining points of the sample. The exact point where analysis is performed (consisting for instance of an inclusion) may be for instance marked in the known manner by the crossing of the wires of a microscope reticle (not shown), the electron beam being directed onto this point by acting upon lenses 13.

According to the invention, the visible or ultraviolet spectrum emitted by a zone or a point having undergone a very high temperature heating under the effect of an electronic bombardment of some milliamperes is obtained, and it is possible to shift from one point or one zone to another of the sample or from one sample to another one by operating crank handle 15.

Finally, in order to replace the samples, plate 20 is unscrewed, the sample magazine is removed from chamber 21 and other samples are fixed on disc 14, after which the magazine is reinserted into chamber 21 and plate 20 is again screwed in position.

The method and device according to the invention have many advantages among which the following ones may be cited:

First, it is possible to perform spectrum analysis in the

far ultraviolet range, therefore to detect elements having a very low atomic number.

It is possible to perform the analysis of gases in the dissolved state.

Analysis point by point of a sample can be effected, which is very interesting in order to study inclusions, segregations and other localized structures.

Finally, it is possible to obtain in a vacuum a stable source, of high brightness and very pure, so as to permit quick analysis.

In a general manner, while the above description discloses what is deemed to be a practical and efficient embodiment of the invention, it should be well understood that there might be changes made in the arrangement, disposition and form of the parts without departing from the principle of the invention as comprehended within the scope of the accompanying claims.

What I claim is:

1. The method of locally analyzing in the visible and ultraviolet range the atomic composition of a material comprising irradiating under vacuum a minute area of said material, by a focalized electron beam of sufficient intensity to locally melt said material in said area and thereby generate a spectrum in the visible and ultraviolet range, representative of the atomic composition of said material in said area, in response to said irradiation, and spectroanalyzing said generated spectrum.

2. The method of locally analyzing in the far ultraviolet range the atomic composition of an alloy, comprising irradiating under a vacuum of about 10^{-4} mm. of mercury a minute area of said alloy by a focalized electron beam of more than one milliamper to locally melt and vaporize said alloy in said area and thereby generate a spectrum in the far ultraviolet range, representative of the atomic composition of said alloy in said area, in response to said irradiation, and spectroanalyzing said generated spectrum.

3. The method of microscopically analyzing a steel sample in the far ultra-violet range for determining the percentage distribution at least of carbon, sulphur, phosphorus, oxygen and nitrogen atoms in said steel sample, comprising irradiating under vacuum a microscopical area of said steel sample by a pulsed focalized electron beam of sufficient intensity to at least melt said steel sample in said microscopical area without producing a material heating of the steel sample around said microscopical area and thereby generate a spectrum in the far ultraviolet range, representative of the percentage presence of said atoms in said microscopical area, in response to said irradiation, and spectroanalyzing said generated spectrum.

4. A spectrometer operating in the visible and ultraviolet range for locally analyzing the atomic composition of solid samples, comprising, in combination, an evacuated vessel, means for supporting in said vessel at least one sample to be analyzed, an electron gun located in said vessel and including a centrally apertured anode and a cathode negatively biased relatively to said anode and arranged to project through said anode a focalized electron beam, means for directing a spot of said focalized electron beam on a determined minute area of said sample, said beam being of sufficient intensity to locally at least melt the material of said sample in said area and thereby generate radiations comprised in the visible and ultraviolet range, representative of the atomic composition of said sample in said area, and visible and ultraviolet radia-

tions dispersing means mounted in said vessel and directed towards said sample to receive said generated radiations.

5. A spectrometer according to claim 4, wherein said means for supporting at least one sample are constituted by a rotatable plate adapted to receive a plurality of samples to be analyzed and by means for rotating said plate so as to bring a determined area of any sample of said plurality in said spot of focalized electron beam.

6. A spectrometer operating in the far ultraviolet range for locally analyzing the atomic composition of alloys, comprising, in combination, a vessel evacuated to a pressure of about 10^{-4} mm. of mercury, means for supporting in said vessel at least one sample of an alloy to be analyzed, a space-charge electron gun located in said vessel and including a centrally apertured anode, a fin-cooled cathode and a D.C. source of over 1500 volts with a positive terminal connected to said anode and a negative terminal connected to said cathode, said cathode projecting through said apertured anode a focalized electron beam forming a spot of minute cross-section on said sample, means for displacing said spot on various areas of said sample, the intensity of said electron beam in said spot being sufficient to locally melt and vaporize the material of said sample in the area covered by said spot and thereby generate radiations in the far ultraviolet range, representative of the atomic composition of said sample in said area covered by said spot, and ultraviolet dispersing means mounted in said vessel and directed toward said sample to receive said generated radiations.

7. A spectrometer according to claim 6 wherein said anode and said positive terminal are grounded and further comprising a control electrode located around said cathode and electrically connected thereto.

8. A spectrometer operating in the far ultraviolet range for determining the local percentage distribution at least of carbon, sulphur, phosphorus, oxygen and nitrogen atoms in a steel sample, comprising, in an evacuated vessel, in combination, means for supporting such a steel sample, a space-charge electron gun including a grounded centrally aperture anode, a fin-cooled cathode, a control electrode located around said cathode and electrically connected thereto, and a D.C. source of over 1500 volts with a positive terminal connected to said grounded anode and a negative terminal connected to said cathode, and therefore also to said control electrode, said cathode projecting through said apertured anode a focalized electron beam forming a spot of minute cross-section on said sample, electron sweeping means for displacing at will said spot on various areas of said sample, the intensity of said electron beam in said spot being sufficient to locally melt and vaporize the material of said sample in the area covered by said spot and thereby generate radiations in the far ultraviolet range, representative of the atomic composition of said sample in said area covered by said spot, and ultraviolet dispersing means mounted in said vessel and directed toward said sample to receive said generated radiations.

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