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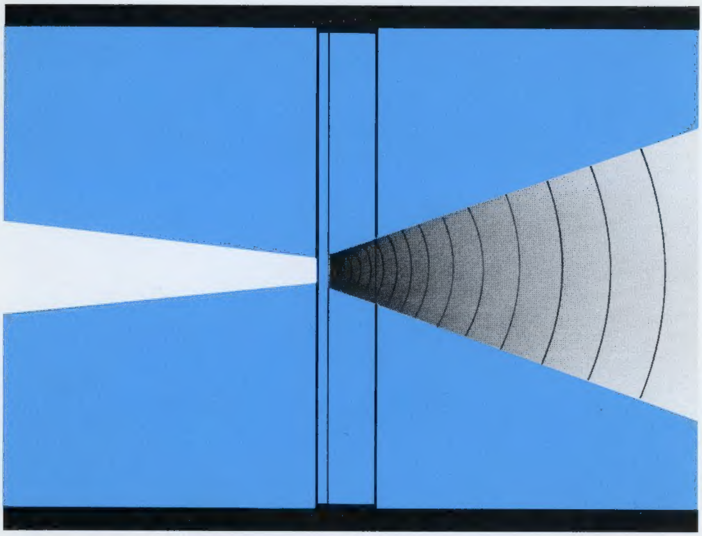
X-Ray Flux from Transmission-Target Tubes



In the past decade, the trend in material analysis has been toward increased accuracy and reduced time required for data accumulation and processing. This demand has created the need for a new X-ray source that can deliver more X-ray flux, is more stable and is capable of being energy and flux tuneable.

Today, radioactive isotopes are still widely used as a stable source of X-rays in elemental analysis because of the isotope's long half-life. However, most of the isotope's radioactivity occurs within a very narrow energy range. As a result, the isotope's monochromatic output limits the chemical analysis to only a few elements. This limitation makes isotopes less desirable where energy and flux tuning is essential to the operation.

Electron bombardment of a target can produce a monochromatic X-ray output, and white radiation known as Bremsstrahlung—the subject of this issue of Tech-notes. Unlike the energy from an isotope, Bremsstrahlung energy is tuneable over a wide spectrum. By tuning the Bremsstrahlung energy, it is possible to selectively excite a specific element directly or indirectly by reradiation (secondary target). Also, the X-ray output flux of an electron bombarded target can be increased readily by increasing the number of electrons striking the target, or turned-off when not in use. X-rays generated by electron bombardment in a transmission-target tube are maintained stable over long-terms, with time periods exceeding 24 hours of continuous operation.

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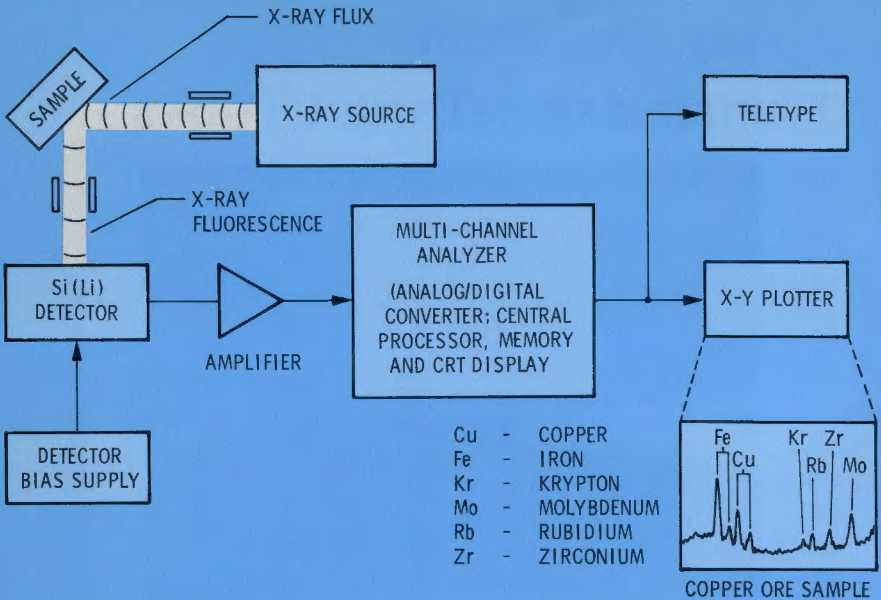


Fig. 1. An energy dispersive analysis system used to detect trace concentrations in a sample by X-ray fluorescence. Under excitation, atoms in the particulate matter sample fluoresce X-rays of different energy levels as shown in the material analysis of a copper ore sample.

Critical Component of Analytical and Process Control Systems

A long-term, stable X-ray source is critical in achieving accurate and repeatable data in quantitative measurement techniques. Typical data collection time may require from a few seconds for a X-ray system gauging sheet metal, up to several minutes for a X-ray energy or wavelength dispersive system measuring trace elements in a chemical composition. In either case, a stable X-ray source eliminates repeated calibration, reduces process monitoring and control interruption, and makes it a valuable tool for analytical research and in-process measurements.

A few of the major applications of automatic analytical or process control systems include:

- Energy dispersive or wavelength dispersive techniques using X-ray fluorescence in chemical composition analysis.
- Diffraction or transmission techniques using X-ray fluorescence in on line manufacturing process analysis and inspection.
- X-ray absorption techniques for on line in-process gauging of material thickness.
- Simultaneous quantitative measurement of content and chemical composition.

Each of these applications requires a long-term constant X-ray output source and is useful in the application of 1) Material Research, 2) Industrial Measurements and Control Systems and 3) Health.

Chemical Analysis by X-Ray Fluorescence

X-ray radiation is produced by incident radiation, i.e. a photoelectric interaction and quantum of energy required to displace an electron from its inner most shell of the atom. This energy appears as Bremsstrahlung (broad energy background) and characteristic

OPERATING CONDITIONS:

DETECTOR	COUNT RATE	COUNT TIME
Si(Li) USED WITH A 1024 CHANNEL X-RAY SPECTRUM ANALYZER	16,180 COUNTS PER SECOND	100 SECONDS PER CHANNEL

CALCULATION OF DATA:

$\bar{x}$	σ	3σ	$\bar{x}+3\sigma$	$\bar{x}-3\sigma$	POINTS OUTSIDE OF $\pm 3\sigma$
1,616,039 COUNTS	1272 COUNTS	3816 COUNTS	1,619,845 COUNTS	1,612,223 COUNTS	NONE

Fig. 2. The long-term stability data recording of a Watkins-Johnson 50 kV, 1 mA transmission-target X-ray source.

lines (monochromatic) of the element. The flux of X-ray radiation is the rate of transfer of X-ray energy across a given surface.

X-ray fluorescence occurs whenever an element is exposed to X-rays. The energy dispersive technique of elemental analysis integrates the emitted X-ray flux over the entire spectrum. The integrations are repeated and the results become the quantitative measurements of the concentration or the purity of an element.

An example of an energy dispersive analysis X-ray fluorescence system used in elemental analysis is shown in Figure 1. Samples of materials under test can be quantitatively analyzed if the X-ray source and other electronic systems are stable over a long sampling period. In basic research of materials, the X-ray source can be used alone or combined with an electron scanning microscope probe to study the physical and chemical properties of an alloy. Gauging a material's thickness and in-process quantitative measurement is accomplished by X-ray fluorescence from or absorbtion of X-ray flux in a material. In both cases, the long-term stability of the X-ray source gives an accurate measure of any change in composition or thickness of material over the length being measured, and assures repeatability of results.

An Example of Long-Term X-ray Stability

X-ray output stability is determined by measuring the amount of change in total radiation over the entire energy range. The photograph of Figure 2 shows the stability data of a 50 kV, 1 mA transmission-target X-ray source extending over a 24-hour period of continuous operation.

A 1024 channel X-ray Spectrometer system counts at a rate of 100 seconds per channel, totals the counts in the spectrum, and then stores the counts in the channel. This process is repeated until all channels (1024) are filled and data in memory is displayed and recorded. From the data, a standard deviation of the mean is computed and can be plotted. The standard deviation (σ) of data points (x) can be defined by the equation:

$$\sigma = \pm \sqrt{\frac{\sum_{i}^{N} (x_i - \bar{x})^2}{N - 1}}$$

where x_i is the i_{th} data point, $\bar{x}$ is the mean of all data points, and N is the total number of data points.

As shown in the photo of data, all data points fall within the $\pm 3\sigma$ limit or ±3816 counts. For an energy dispersive system, the results shown mean that all data points will lie within a statistically predicted deviation from the mean. Thus, the source does not introduce any error into the quantitative analysis.

X-Ray Flux Generation

The total X-ray flux generated by a X-ray tube consists of the continuous spectrum referred to as Bremsstrahlung, and monochromatic chracteristic lines (K, L, M, etc) of the target material.

These characteristic X-ray lines are produced when the incident electron energy is equal to, or greater than, the energy required to displace an electron from the inner most shell of an atom. Figure 3 is a diagram showing an example of the X-ray spectrum of a target made of a single element (only $K\alpha_1$, $K\beta_1$, $L\alpha_1$ and $L\beta_1$ lines are shown.)

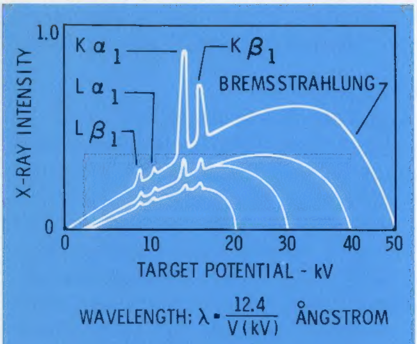


Fig. 3. The spectrum of X-rays produced when an electron beam strikes a target made of a single element.

The continuous spectrum cutoff wavelength depends on the accelerating potential of the electron beam and is independent of the target material, while the position of the characteristic line peaks ($K\alpha_1$ and $K\beta_1$) depend on the target material and are independent of the accelerating potential. The characteristic lines $L\alpha_1$, and $L\beta_1$ and other peaks appear at longer wavelengths (lower KeV) corresponding to each electron shell.

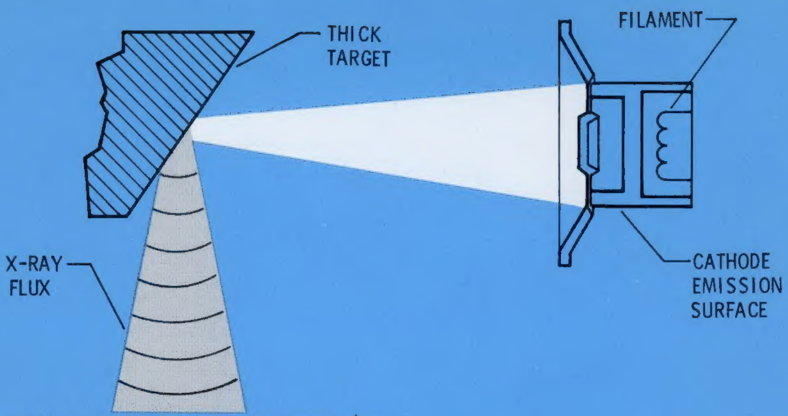
The total radiation emitted, whether by Bremsstrahlung or by a characteristic nature, can be expressed by the approximate intensity equation:

$$I_{int} = A\, Z\, i\, V^2$$

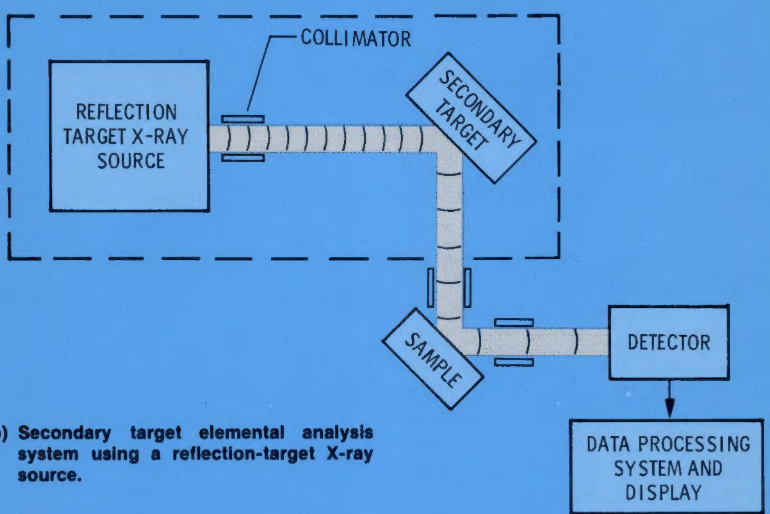
where A is a constant approximately equal to 1.4×10^{-9}, Z is the atomic number of the element, i is the electron beam current in amperes, and V is the target potential in volts. Thus, the X-ray intensity for a particular target material depends upon the electron beam current and the voltage applied to the target.

Reflection Type X-Ray Radiation—Thick Target Tubes

X-ray radiation generated by an electron bombardment of a target is energy and flux tuneable by electronically varying the target voltage and current respectively. Conventional X-ray generators, Fig. 4a, use directly and indirectly heated cathode electron sources. A directly heated filamentary cathode operates in the temperature limited region of electron emission and is sensitive to temperature changes. An indirectly heated cathode (shown in Figure 4a) operates in the space charge region and is much less sensitive to changes in



a) X-ray flux of a reflection-target tube containing an indirectly heated filamentary cathode.



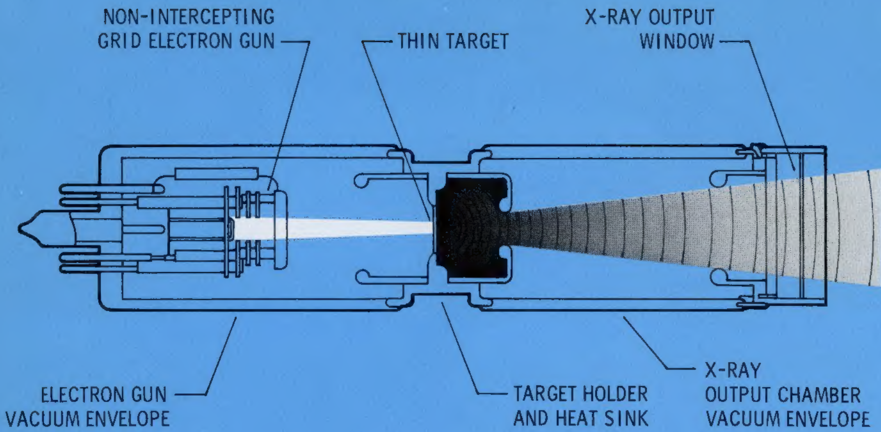
b) Secondary target elemental analysis system using a reflection-target X-ray source.

Fig. 4. X-ray fluorescence generated by a reflection-target X-ray source and a commonly used secondary target elemental analysis system.

temperature. Electrons emitted from an indirectly heated cathode do not depend primarily on the filament's thermal characteristics.

Until recently, the most common X-ray fluorescence excitation has been generated by a reflection type (thick target) X-ray tube. Along with this thick target tube, a secondary target or heavy filtering is required to reduce interference inherent in Bremsstrahlung radiation for analysis of trace elements, Fig. 4b. Because of the geometry used in the excitation of the secondary target system, the overall efficiency of the X-ray output is extremely low, approximately 0.01 percent. This poor efficiency demands the use of high power tubes that usually require liquid cooling; moreover it increases the sytem's complexity and operating cost.

A transmission-target tube recently developed at Watkins-Johnson provides an alternate way of achieving



a) X-ray flux of a transmission-target tube containing a non-intercepting grid electron gun.

Fig. 5. X-ray fluorescence generated by a transmission-target X-ray source and a direct elemental analysis system.

the desired detection signal level with much less electron beam power consumption.

Transmission Type X-Ray Radiation—Thin Target Tubes

The first known transmission-target (thin target) X-ray tube was built as a laboratory instrument for chemical analysis using X-ray fluorescence[1]. The elemental analysis performed using this type of tube was comparable to a secondary target system, however, the tube used less than 1/10th of the electron beam power required for a secondary target system. The transmission-target tube, Fig. 5a, consists of a grounded cathode non-intercepting grid electron gun, a transmission-target at elevated potential and a X-ray flux output window. The thin target generates characteristic X-rays which produce less Bremsstrahlung and which provides additional self filtration.

Because the characteristic lines of the transmission-target tube are generated with less background noise, the tube is used in a non-reradiating elemental analysis system shown in Figure 5b. In this direct analysis system, X-radiation losses are reduced due to the reduction in distance from sample to primary source. By eliminating the reradiation of X-ray flux, the efficiency of the direct analysis system is increased. A direct consequence of the higher efficiency is the reduction of beam current and power.

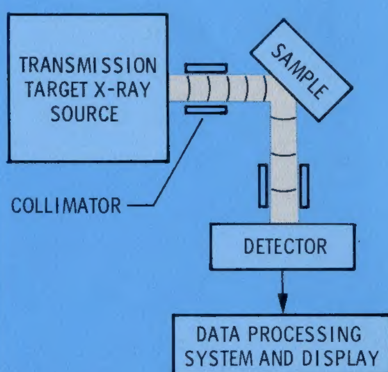


Fig. 5b. Direct elemental analysis system using a transmission-target X-ray source.

[1]Reference No. 1.

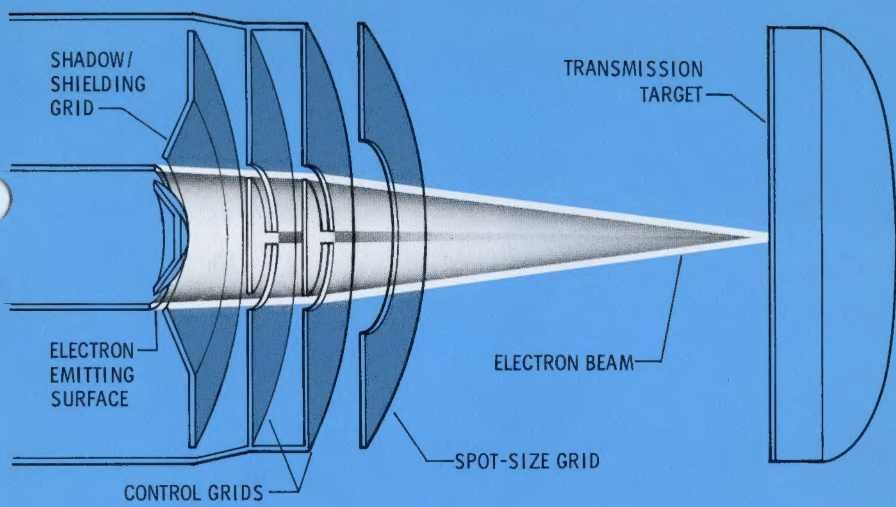


Fig. 6. A cross-sectional view of Watkins-Johnson's non-intercepting grid electron gun and electron beam.

Since the secondary target system requires farther distances, only a small amount of radiated energy from the secondary target is useful in reradiating the sample under test. By direct reradiation of the sample, the additional amount of X-radiation which would be required of the secondary target system is conserved. As a result, the size and power requirements of the transmission-target tube are reduced. In many automatic analysis systems, this reduction in tube size decreases the operating cost of the system. Smaller X-ray sources can be physically placed closer to the sample under test, thereby increasing the X-ray intensity at the sample. For example, X-radiation varies as the square as the distance from the source, a decrease in distance by 1/2 results in a 4 times increase in X-ray flux at the sample.

Non-Intercepting Grid Electron Gun

The electron gun[2], Fig 6, is best characterized as a shadow-grid gun, that is, the grids are stacked in the shadow of the previous grid closest to the cathode. A different potential is applied to each grid, usually increasing in magnitude from the cathode. The grid potentials force the electrons to pass through the orifice in the grids, producing a hollow cylindrical shaped electron beam. The design of the cathode emitting surface and grid structure yields an extremely low electron beam interception. In recent X-ray tubes the grid interception is less than 0.1 percent, however, a 0.01 percent interception is achieved by a non-intercepting grid gun with -15 volts applied to the control grids.

Electron emission (beam magnitude) from the cathode is controlled by control grids; the spot-size grid controls the size of the electron beam area at the target. Because of unique gun optics, the target potential has no influence upon the electrons emitted from the cathode, and only the control grids are used to turn-on or turn-off the X-ray output and to maintain a preset and constant level.

The spot-size grid is the key com-

[2]Reference No. 2.

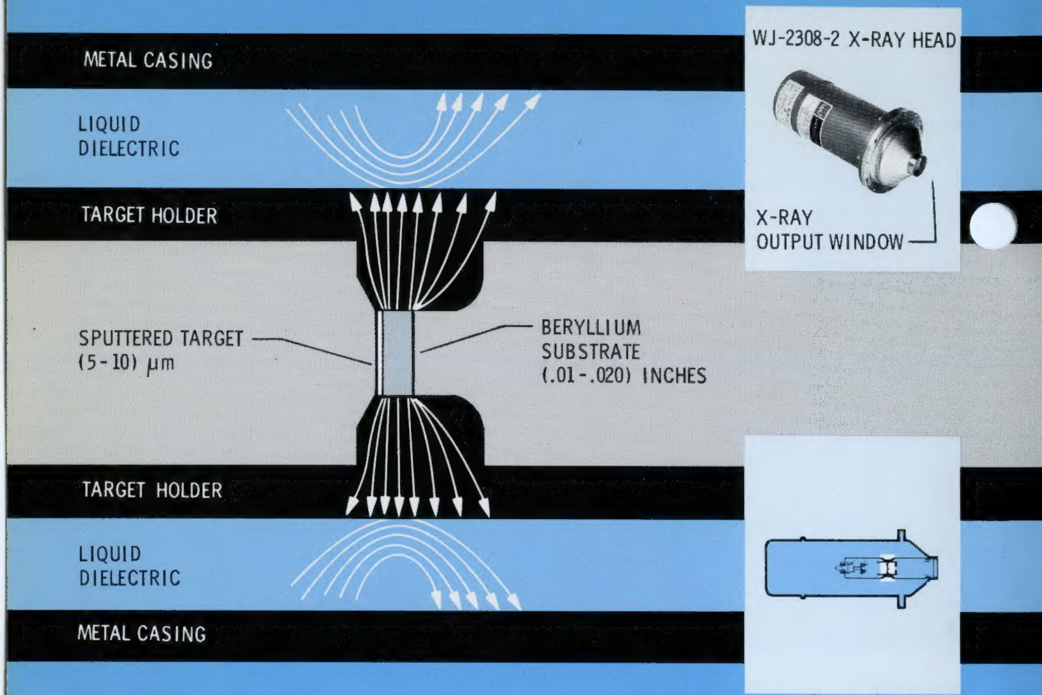


Fig. 7. A cross-section of the transmission-target and critical heat transfer region within the transmission-target X-ray source. Actual size of the WJ-2308-2 X-ray head which contains the transmission-target tube is 2-7/8 inches in diameter.

ponent in adjusting and maintaining a constant beam spot size in order to prevent burn out of the thin target at full power operation. Besides controlling the size of the beam spot at the target, the spot-size grid provides additional focus for the beam-edge to prevent primary electrons from bombarding and charging the glass envelope. Charging the glass at a potential of the beam causes an instability in the beam spot position and produces an unstable X-ray output. By using a computer program[3], Watkins-Johnson determined the shape of this angled electrode, resulting in optimum beam-spot stability.

Critical X-Ray Source Region—The Transmission-Target

The target is a collector of electrons, therefore, it is necessary that the target transfer heat efficiently. Transmission-targets are made from either a thin foil, or from the desired elements deposited on a Beryllium substrate, Fig. 7. By brazing a 0.002-0.003 inch thick transmission-target foil to a Molybdenum target holder, maximum heat transfer is achieved. A typical deposited target thickness is 5-10 microns (μm) and is sputtered on a 0.010-0.020 inch thick Beryllium substrate.

In order to dissipate heat, the tube is enclosed by a liquid dielectric which conducts heat from the target holder to the metal casing of the X-ray head. Beryllium increases the heat dissipation capacity of the tube by allowing the use of a thicker substrate due to its low attenuation to X-ray flux. Materials used for sputtering are of high purity and some of these elements include: Tungsten, Molybdenum, Chromium, Gold, Silver, Rhodium and Copper. Target deposition by sputtering

[3]Reference No. 3.

techniques permits only the exact amount of an element to be deposited onto the substrate, thus reducing X-ray flux losses due to target attenuation.

The thin-foil target provides excellent filtering of Bremsstrahlung, however, it also reduces the total available flux and is therefore less efficient. Because the foil is made thin to reduce attenuation of the available flux, its heat transferring capacity is less than the sputtered target on Beryllium substrate.

Spectrum Purity of Transmission-Target X-Rays

Spectral purity is essential in analyzing or searching trace elements in a chemical composition. This analysis is ordinarily performed by exciting characteristic X-rays of an element with the energy of another monochromatic X-ray that is higher than that required to remove an electron from the inner shell(s) of the element under investigation.

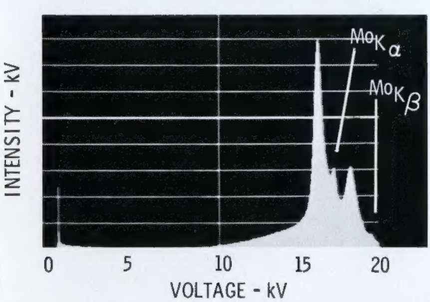
Figure 8a shows the spectral lines emitted from the Molybdenum (Mo) target in a transmission-target X-ray tube. Using these characteristic lines, a filter paper sample is analyzed for contaminants, Fig. 8b. The spectral purity achieved, shown in Figure 8b, is comparable to that of a secondary target tube source, and is primarily due to the attenuation of background radiation inherent in the 0.0022 inch Molybdenum transmission-target. This high degree of spectral purity is achieved by using pure Molybdenum parts, the construction techniques used in shielding brazed joints, and shielding tube parts from the incident electron beam and non-collimated output flux.

X-Ray Source Stability—Electrical and Mechanical Parameters

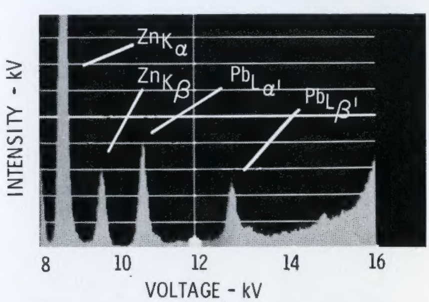
The parameters affecting the stability of a transmission-target source are both electrical and mechanical.

- **Electron Beam Control.** X-ray flux stability depends on the accelerating electron beam potential and the quantitative control of target current. X-ray flux stability is achieved by keeping the number of electrons striking the target constant. By controlling the beam current with a closed loop circuit, the circuit regulates the target current as a function of grid voltage.

A change in the electron beam area or position at the target is reflected as a change in X-ray output flux. A 2 percent change in beam position or size can result in an equal amount of change in X-ray flux at the detector. Thus, the spot-size



a) Emission spectrum of a Molybdenum target in a WJ-2380 transmission-target X-ray source.



b) Emission spectrum from a filter paper sample by using the radiation of the characteristic lines from the Mo target source. Trace concentrations of zinc (Zn) and lead (Pb) are shown detected to approximately 10 parts per million.

Fig. 8. X-ray emission spectrums of a) a Molybdenum target and b) a material sample.

control grid voltage must maintain the electron beam at the target constant.

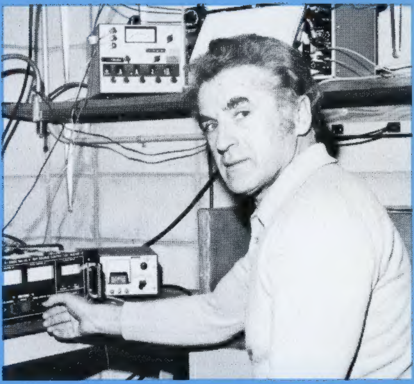
Because the X-ray intensity is proportional to the square of the target voltage, any change in voltage will affect stability. In order that the X-ray output flux be within an established ±0.2 percent deviation from the accumulated data points, the target voltage is load regulated to less than 0.05 percent.

• **Spatial Motion.** Spatial motion of tube components will affect the stability of the output flux. Of all the tube components, target motion is the most critical. Mechanical parameters are affected by heat dissipation in the target. By transferring heat from the point of electron impact to the heat sink, Fig. 7, mechanical variation in target-to-window spacing is minimized. The X-ray flux radiated from the tube's output window is dependent upon the square of the distance from the target. For example, a 2 percent change in target-to-window spacing will produce a 4 percent change in X-ray flux at the output window. In practical analytical systems, X-ray stability must be maintained to less than 0.05 percent.

Summary

X-ray intensity is a function of the combined design characteristics of target voltage, target current and mechanical parameters. Control of these characteristics has achieved a stable X-ray output intensity with a coefficient of variation ($\sigma/\bar{x} \times 100\%$) less than ±0.2 percent. The ±0.2 percent coefficient of variation is achieved by utilizing advanced electronic techniques in the W-J source control unit and transmission-target X-ray source. This result is comparable to the results obtained with a radioactive isotope, the standard in the industry today. However, the Watkins-Johnson X-ray source has the additional benefit of versatility by tuning X-ray energy and flux.

Author: William Hershyn



William Hershyn earned his B.S.E. E. at the Massachusetts Institute of Technology and became a member of the technical staff at W-J's Stewart Division in 1967. Mr. Hershyn is currently Head, X-ray Development responsible for R & D of X-ray sources and equipment for analytical and general commercial use. Mr. Hershyn is responsible for the development of the 50 kV, 1mA transmission-target and reflection-target tubes (also the 2 mA version of both), 110 kV peak, 200 mA (pulsed) X-ray source and the electronic feedback loop circuitry to control target current; the necessary power supplies and control circuits. Mr. Hershyn's previous responsibilities inciuded: Project Engineer for the development of the electron beam bombarded semiconductor pulsed Class B, RF amplifier, and as Program Manager for the development of gain matched, medium noise, quad amplifiers used in the guidance of the Shrike Missile. Bill is a member of the IEEE.

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