

X-Ray Fluorescence Metrology Tools

Technology
Guide



Introduction

1. Introduction
2. X-Ray Fluorescence
3. XRF Analysis System
4. X-Ray Generation
5. X-Ray Collimation
6. X-Ray Detection
7. Pulse Processing
8. Spectrum Processing
9. Quantitative Analysis
10. System Performance
11. X-Ray Beam Targeting
12. Software & Interface
13. Glossary

About This Guide

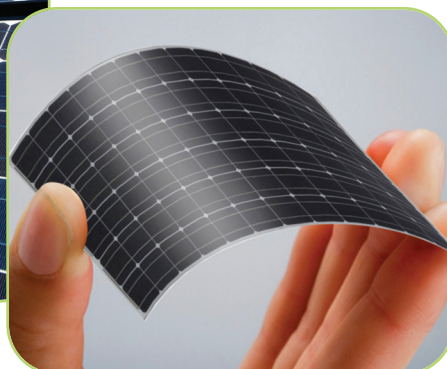
The purpose of this guide is to familiarize the prospective buyer and/or current user with the fundamentals of energy dispersive X-ray spectrometry, commonly referred to as X-Ray Fluorescence (XRF).

XRF analysis is a powerful quantitative and qualitative tool ideally suited to the analysis of film thickness and composition, determination of elemental concentration by weight of solids and solutions, and identification of specific and trace elements in complex sample matrices.

XRF analysis is used extensively in almost all industries, including semiconductors, photovoltaics and renewable energy, power storage, telecommunications and microelectronics, metal finishing and refining, food, pharmaceuticals, cosmetics, agriculture, plastics, rubbers, textiles, fuels, chemicals, and environmental analysis.

The wide deployment of XRF analysis tools in industry is the result of XRF's ability to perform non-contact, non-destructive tests with speed and precision, combined with low cost of ownership.

This guide is designed to be brief. Each section examines one aspect of the overall application of XRF for the analysis of matter. Emphasis is given to the measurement of film thickness and the quantitative determination of element concentrations in solids, and film thickness/composition of deposited film alloys.



When a material is exposed to a source of X-rays, two primary interactions between the X-rays and the atoms composing that material take place: (1) X-ray scattering and (2) the photoelectric effect.

Of the two primary interactions, the photoelectric effect is the most interesting from the point of view of providing information about the exposed material. The photoelectric effect in this case results in a process known as X-ray fluorescence. By definition, fluorescence is a process whereby a material absorbs energy and almost instantaneously releases energy in a characteristic form.

On a microscopic level, all atoms can be represented by a positively charged nucleus surrounded by a "cloud" of negatively charged electrons (figure 2). Electrons occupy specific energy levels (orbitals) in a configuration around the nucleus which is unique for each element in the periodic table.

The electrostatic attraction between each electron and the positively charged nucleus maintains the electron's position within the atom relative to the nucleus. The potential energy for each electron depends on its orbital position. Only specific, discrete energy levels may be occupied by any electron for a given element.

When exposed to X-rays with sufficient energy, a finite probability exists that an inner orbital (usually first and sometimes second or even third "shell") electron will absorb that energy. With this added energy, the bond between the electron and the nucleus is broken and the electron leaves the atom.

After an inner orbital electron is ejected from an atom, an electron from a higher orbital (with higher potential energy) will drop down to fill the lower orbital vacancy. The resultant change in potential energy is expressed by the release of an X-ray photon. These photons are detected by the XRF instrument.

The potential energy between two given orbitals of a specific element is exact and is always the same. Likewise, the X-ray photons emitted when an electron transitions from one orbital to another always have the same energy (that is, the same frequency and wavelength). The potential energy difference between two given orbitals of any element is unique to each element in the periodic table. For this reason, X-rays emitted by the photoelectric effect are called characteristic X-rays.

Typically, with respect to X-ray fluorescence, there are from two to several (more than 10) orbital transitions possible within a given atom, depending on its atomic number and resultant electronic configuration. Therefore, each element will emit a unique series of specific X-ray energies (line spectrum). By determining the energies of the photons detected from an unknown material, it is possible to determine the identity of the elements composing that material.

When a sample is exposed to an X-ray source with a known, constant intensity, it will fluoresce characteristic X-rays at a rate (intensity) proportional to the concentration of the element(s) present in the sample. It is therefore possible to quantitatively measure the thickness as well as the elemental composition of the sample.

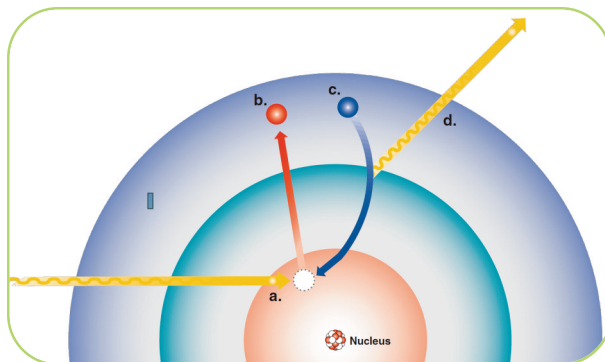


Figure 2: X-Ray Fluorescence

- a. An incoming X-ray photon strikes an electron orbiting the atomic nucleus.*
- b. The electron breaks free of its orbit and leaves atom.*
- c. An electron from a higher energy orbital replaces the freed electron (electron cascading).*
- d. As it drops to the lower orbital, the electron releases energy (fluoresces), in the form of an X-ray photon, the energy of which is equal to the potential energy difference between the higher and lower energy orbitals.*

The basic design of any XRF thickness and composition metrology tool incorporates five features:

- A source of high intensity X-rays.
- A collimation apparatus to define the X-ray beam size.
- X-ray beam targeting and sample positioning mechanisms.
- X-ray detection, processing, and analyzing electronics.
- Algorithmic tools for determining the thickness and/or composition of the sample.

How It Works

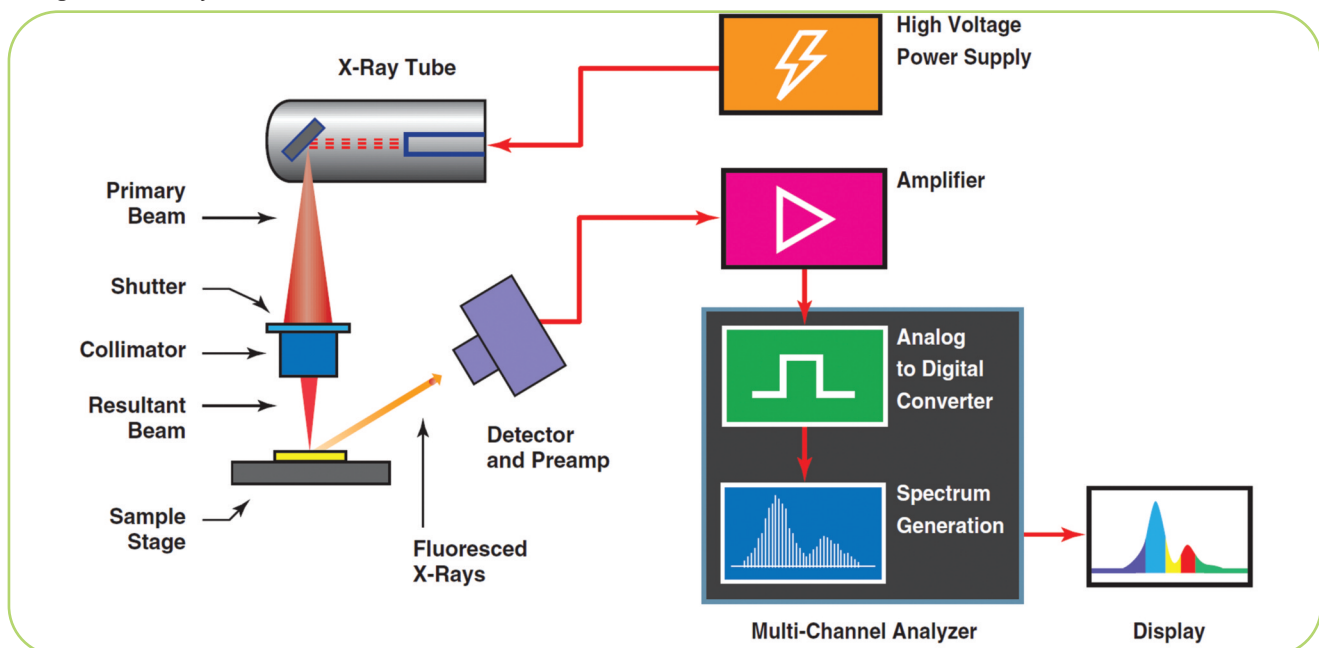
When an element is exposed to a source of high intensity X-rays, it will emit X-rays (fluoresce) at energy levels unique to that element.

The XRF tool uses a high intensity X-ray tube to generate X-rays. These primary beam X-rays are directed through a collimator and emerge in a tightly focused resultant beam with a specific cross-sectional geometry. The resultant beam impinges the sample material and induces fluorescence from the elements present. A detector senses X-ray emissions from the sample and converts them into a series of analog pulses. The amplitude of each pulse is proportional to the energy of each incident X-ray.

An amplifier shapes the analog pulses into workable electronic signals (0 - 5V). These signals are converted into digital values by an analog-to-digital converter (ADC). The digitized pulses are sorted according to energy level and stored in a memory location (channel) by a multi-channel analyzer (MCA).

The number of pulses stored in each channel are counted to generate a frequency distribution (histogram), displaying channel numbers along its X axis (corresponding to energy level), and counts (the number of pulses detected at each energy level) along its Y axis. This histogram is known as a spectrum or pulse height analysis (PHA), and is the raw data from which thickness and composition determinations are made. The XRF employs a combination of mathematical techniques to further refine the raw spectral data into a pure spectrum. The pure spectrum is correlated with a calibration model to produce a measurement result.

Figure 1: XRF System Schematic



The XRF tool induces fluorescence from a sample by exposing it to a source of high intensity X-ray radiation.

A typical X-ray source consists of an evacuated glass tube containing a filament cathode (typically tungsten) and a quantity of target anode material. The tube is sealed in an oil-filled metal (typically brass) enclosure that effectively shields X-rays.

When a current is applied to the filament, it emits negatively charged electrons which are accelerated toward the target material by a high voltage potential. These electrons interact with the target material to produce a broad spectrum of X-rays. These primary beam X-rays exit through a “window” in the tube enclosure and are directed down the generation conduit into the collimation mechanics.

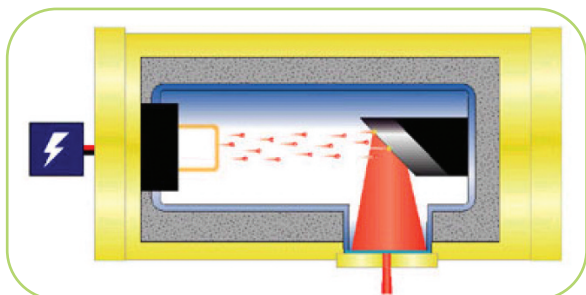


Figure 3: X-Ray Generation

- Current applied to the filament liberates electrons into free space within the tube.
- Electrons are accelerated toward the target by an electric field produced by a high voltage power supply.
- Electrons interact with the target to produce primary beam X-rays (see detail, figure 4).
- Diagram at right shows primary beam X-ray output from Molybdenum (Mo) target tube. The Mo target produces a polychromatic X-ray spectrum capable of exciting fluorescence from a broad range of elements.
- A primary mechanical filter may be positioned between the primary beam and the sample to selectively block some of the primary beam X-rays, modifying the output of the source to enhance the excitation of certain elements of interest.

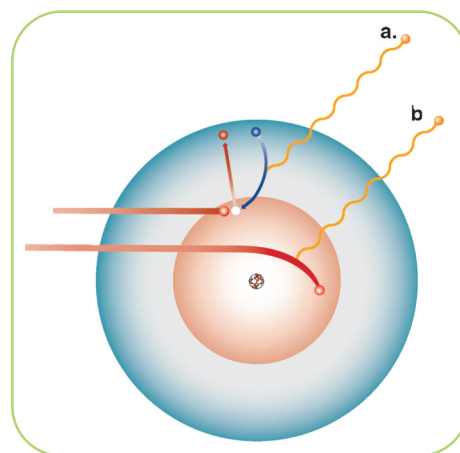
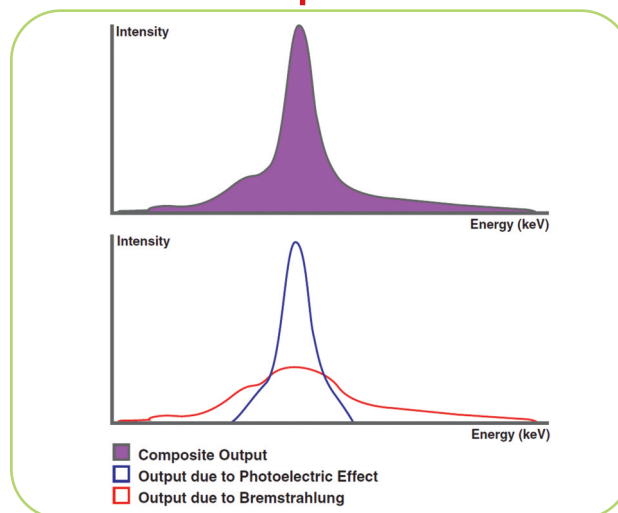


Figure 4: Filament/Target Interaction

Interaction between accelerated filament electrons and target atoms produces fluorescence by means of two processes:

- Photoelectric Effect:** Filament electrons transfer energy to target atom electrons, causing ejection of those electrons from their orbitals and a resultant photoelectric emission (fluorescence).
- Bremstrahlung:** Literally, “braking radiation”. Filament electrons are attracted to the strong positive charge of the nuclei, causing deceleration and release of energy.

Primary Beam X-Rays



The consistent, controlled, and well-defined X-ray beam geometry is critical to measurement accuracy. The process of shaping the primary beam geometry is called collimation. There are two principal methods of collimation, mechanical and optical.

Mechanical Collimation

A mechanical collimator is essentially a pin-hole aperture. A typical mechanical collimator assembly consists of a metal block featuring collimators of various dimensions (figure 5). To form the collimated beam, a single collimator is aligned directly beneath the path of the primary beam X-rays. X-rays pass through the collimator and emerge in a resultant beam whose initial dimensions are equal to the dimensions of the collimator and whose final dimensions fan out to a larger geometry. This resultant beam is targeted at the sample.

Optical Collimation

Optically collimated XRF tools employ an optical element to shape and direct primary beam X-rays. Primary beam X-rays incident upon the surface section of the optical element (figure 6) propagate through the system and emerge in a convergent beam of extremely high intensity.

Advantages and Disadvantages

Mechanical collimators form the incident beam by blocking all but a small percentage of the primary beam X-rays (those traveling in a path coinciding with the orientation of the collimator tube). Mechanical systems are currently less expensive and provide more than sufficient incident beam intensity for a wide range of analytical tasks where small beams (under 4 mils (100 microns)) are not required.

Optically collimated systems capture and transmit a substantially higher quantity of the primary beam X-rays produced (100 to 1000 times that of mechanically collimated systems). A higher intensity beam produces significantly higher X-ray countrates from the sample, with commensurate benefits to precision. XRF tools utilizing optical collimators are referred to as Microbeam XRF (MXRF) tools.

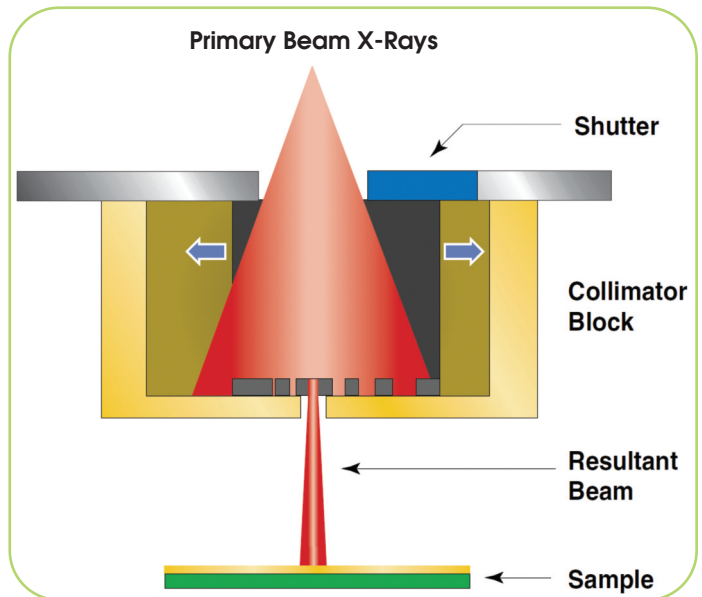


Figure 5: Mechanical Collimation

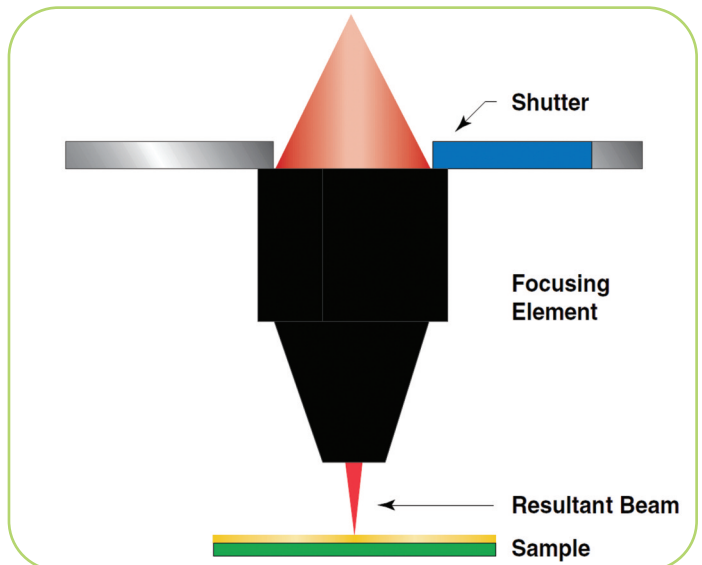


Figure 6: Optical Collimation

Once a sample has been excited to fluorescence, a detector is used to collect and convert X-rays into electronic signals which can be used to determine energy and intensity (number of X-rays) emitted from that sample. This is sometimes referred to as energy dispersive X-ray detection. There are two commonly used types of energy dispersive detectors, the proportional counter and the semiconductor detector.

Proportional Counter Detectors

The proportional counter is a sealed, gas filled hollow cylinder with a central coaxial wire. An electric field is established between the outside shell and the inner central wire using an external high voltage supply. X-rays enter through a window (usually thin beryllium) in the detector shell and ionize the gases inside. Electrons are attracted to the positively charged wire, producing a charge at the detector output. This charge is collected through a pre-amplifier and converted into a voltage pulse, which is then further amplified and shaped by an amplifier. The resultant pulse amplitude is directly related to the number of ion pairs created by the ionizing X-ray photon, which in turn is directly related to the energy of the incident photon.

Semiconductor Detectors

The two most common semiconductor detectors are the PIN Diode and Si(Li) types. Both detectors incorporate silicon chips which respond to X-rays in the same manner as the gas in the proportional counter.

The pin diode is maintained under a reverse voltage bias, leaving a central p-n junction where detection of X-rays can occur. The Si(Li) semiconductor also provides a p-n junction for X-ray detection, except that the thickness of its sensitive region is increased through the use of a process known as "lithium drifting". Incident X-rays produce ionizations of Si in the sensitive region of these detectors. The charge carriers

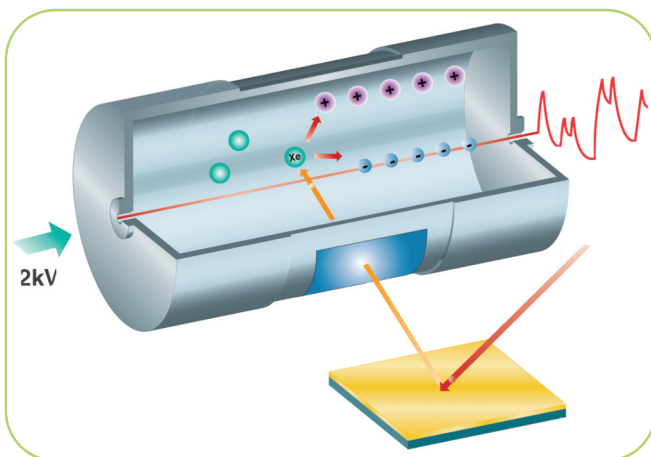


Figure 7: Proportional Counter Detector

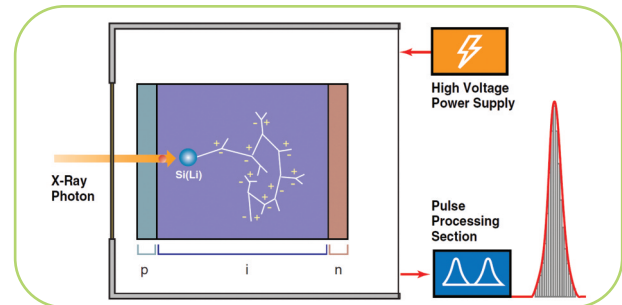


Figure 8: Semiconductor Detector

are negative electrons and positive "holes", which are drawn to opposite ends of the detector due to the voltage bias applied across the silicon chip. Like the proportional counter, total charge collected within the semiconductor detector is directly proportional to the energy of the incident X-ray, and is converted to a corresponding voltage amplitude through the use of a preamplifier and amplifier.

To minimize electron/hole creation due to thermal effects (ensuring that pulses are produced in response to X-ray ionizations only) semiconductor detectors often employ cooling units to maintain very low operating temperatures. Typical cooling schemes use liquid nitrogen or electric heat exchange.

Relative Advantages of Each Detector Type

Proportional counters are less expensive, require no maintenance, and are very robust detectors. Their large volumes and window surface areas provide a large solid angle for X-ray capture. The result is higher capture rates, providing higher intensity measurements (resulting in better measurement precision). The detector gas is more sensitive to higher energy X-rays, making proportional counters more efficient for detection of X-rays from high concentrations and/or thick layers of Sn, Pd, Ag, Cd, In and Rh.

Semiconductor detectors provide superior resolution. Detector resolution is influenced by electronic noise and the statistical variation inherent in the creation of ion pairs. Compared to proportional counters, semiconductor detectors produce many more pairs of charge carriers per X-ray event (for X-ray energies which are equal), reducing statistical variation. The result is an improved ability to distinguish X-ray energies which are similar. Si(Li) detectors offer better resolution than the pin diode, and better sensitivity for higher X-ray energies since the sensitive region is thicker.

Charges produced in response to ionization in the detector are fed through a preamplifier and emerge from the detector output in a form similar to that shown in figure 9a. A series of additional pulse processing operations are performed to transform the initial preamp pulse into usable information about the X-ray energies and intensities detected.

Pulse Processing

The preamplifier in the XRF acts as an interface between the detector and pulse processing circuits. It is a charge sensitive device, converting charges collected by the detector into a voltage.

An amplifier further amplifies the preamp output voltages and gives them a pulse shape. It is also designed to minimize noise from the preamp. A typical output pulse from the amplifier might look like that shown in figure 9b.

Amplifier pulses are sent to a multi-channel analyzer (MCA), which converts the analog pulses into digital signals (figure 9c). Each digital value is subsequently sorted and stored in a channel in the MCA. A channel is a memory location representing a small range of energies. As each pulse is digitized, it is stored in a channel corresponding to its amplitude (representing an X-ray energy level), and a counter for that channel is incremented by 1.

An XRF measurement represents the detection of X-ray events over some finite, preset time period. The end result of a measurement is a collection of stored digital signals sorted by pulse height. These values are displayed graphically as a frequency distribution (histogram of energy vs. intensity), referred to as the spectrum (figure 9d).

At all stages in the pulse processing chain, proportionality between the detected X-ray energy, the analog pulse

amplitude, the digital signal value, and the corresponding channel number is strictly maintained.

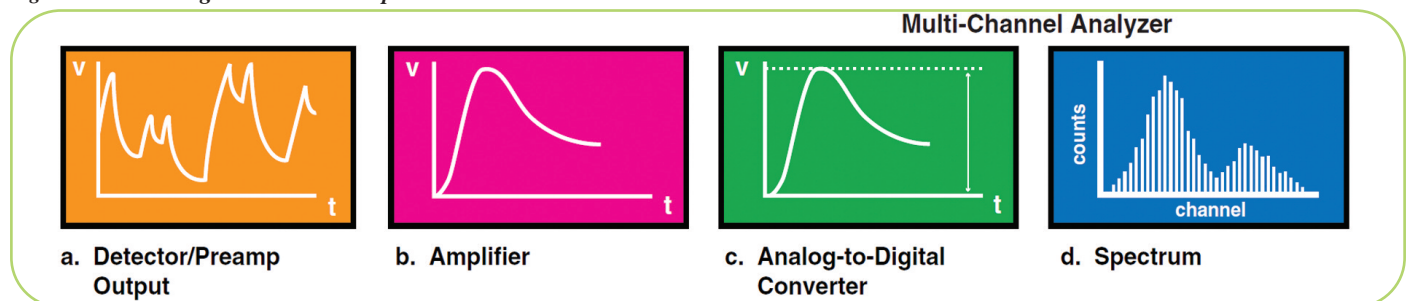
The processing of individual X-ray events occurring at widely spaced time intervals is a relatively straightforward matter. However, real world X-ray events occur at randomly spaced intervals. Since a finite period of time is required to detect a pulse and process its signal, some errors in pulse processing may occur, depending on the interval between pulses. For example, while the electronic signal resulting from one X-ray detection is being processed, another signal may be created by a new X-ray event before the first signal has been completely evaluated. Such errors can become more pronounced as X-ray intensity (the rate at which X-ray photons enter the detector) increases. To compensate for these factors, corrections are designed into the pulse processing sections of the XRF tool. These include dead time correction, pulse pile up rejection, and baseline restoration (see glossary).

The Spectrum

The digitized pulses, or counts, generated by an X-ray emission from a given element (whose characteristic energies are exact) will be observed as a peak within a distribution of counts surrounding the channel containing the highest number of counts (peak channel). This is because:

- Statistical variations occur in the number of ionizations produced in the detector by X-ray photons of the same energy (see figure 10).
- The resulting analog signal is superimposed over a randomly varying level of electronic noise, or background (see figure 13).

Figure 9: Processing the Detector Output

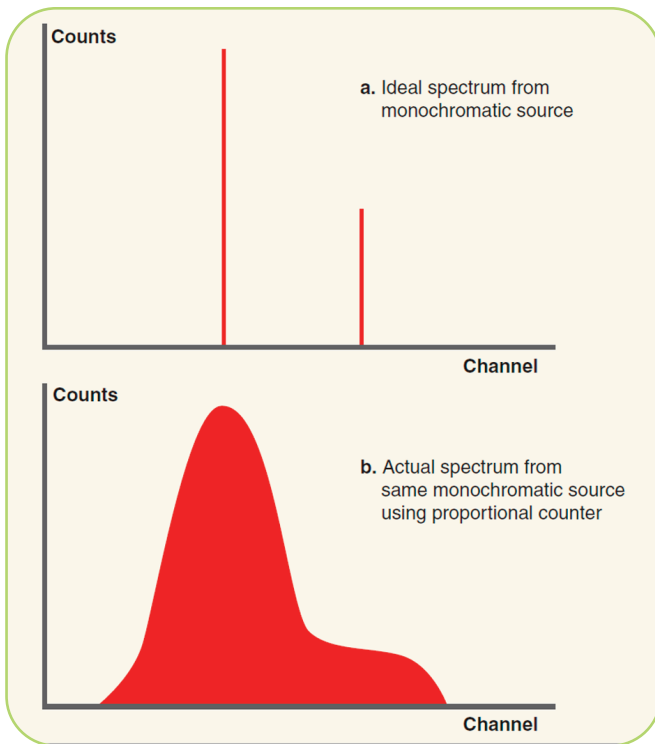


Pulse Processing

Qualitatively, the width of this distribution describes the combined resolution of the detector and pulse processing electronics.

From the resultant spectrum (figure 11), X-ray energy level, and therefore elemental, identification is accomplished by determining the channel numbers of the observed spectral peak(s). Quantitative information (thickness/composition) about the element(s) of interest may be derived by determination of the peak height(s) or integration of the spectral peak to determine overall peak area, which represents the number of X-rays detected for a given energy.

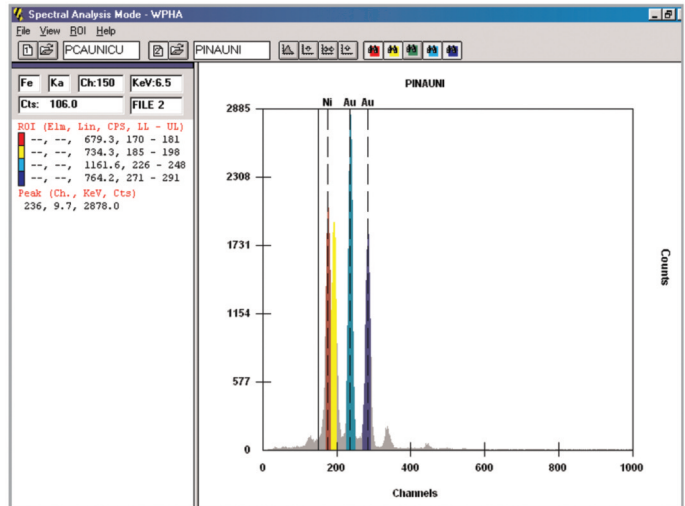
Figure 10: Distribution of X-ray counts



In figure 11 spectral peaks are produced by high X-ray counts at specific energies. In the pulse height histogram (spectrum), X-ray counts are displayed on the vertical (Y) axis. Energy level ranges (channels) are displayed along the horizontal (X) axis.

Semiconductor detectors are capable of generally superior energy level resolution compared to proportional counter detectors. As a result, the peaks exhibited by semiconductor detector spectra (top) will comprise a narrower band of channels than those obtained using a proportional counter (bottom) when measuring the same material(s).

Film Stack Sample Spectrum, Panel 3786



Film Stack Sample Spectrum, Panel 3786

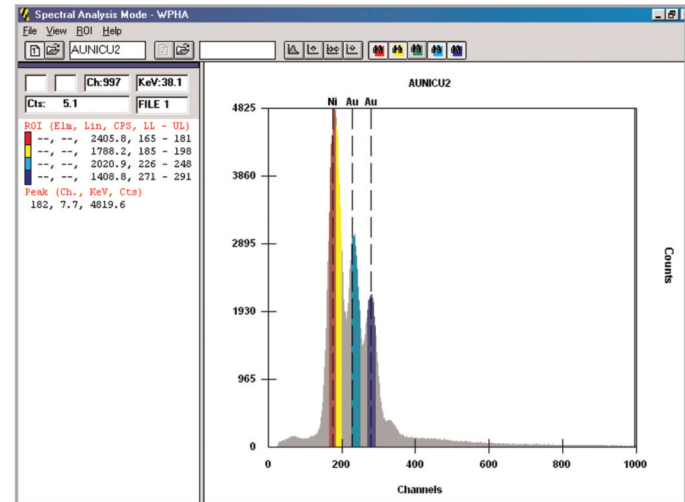


Figure 11: Pulse Height Histogram (Spectrum)

Spectral peaks produced as the result of excitation of various elements in the sample may be quantified. The most common approach to quantifying spectral data is to integrate a peak over a given range of channels, or Region of Interest (ROI). Typically, the lower and upper limits of the ROI define a band of channels where pulses collected (counts) are significantly higher than in the channels surrounding the peak; such background channels are characterized by low count levels caused by X-ray scattering from the sample and artifacts of the pulse processing electronics.

While accurate quantitative determinations can often be made by simply integrating over the ROI limits, it is sometimes necessary to further process the spectrum. A combination of mechanical and mathematical methods are used to refine spectral data, including:

Peak Deconvolution (Numerical Filtering): Peak overlap is observed when the characteristic X-rays detected from two or more elements are close in energy. The degree of overlap depends on the similarity of the energies and on the resolution of the detector used. Peak deconvolution is employed to mathematically decompose two or more peaks which overlap and superimpose to produce a single, wider composite peak (figure 12). A variety of techniques are available for peak deconvolution, with advantages and limitations inherent in each method depending on the application being analyzed.

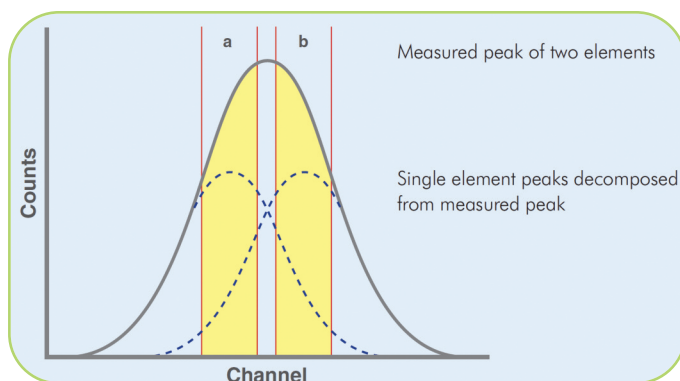


Figure 12: Peak Deconvolution

ROIs (a) and (b) are integrated to solve the deconvolution functions once pure element reference spectra are measured and stored.

Background Correction: Spectral peaks are initially superimposed over a background level of counts (figure 13). These readings originate from X-ray scattering, electronic noise, and errors in pulse processing. Various mathematical

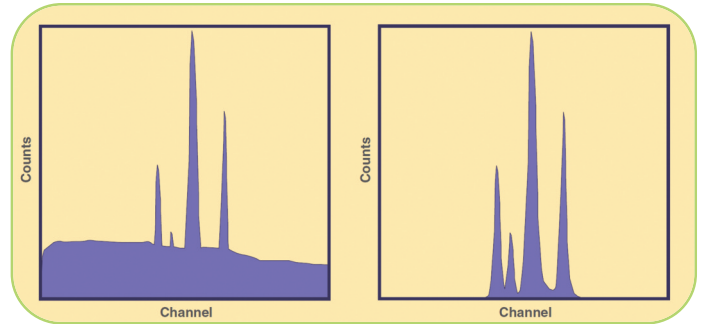


Figure 13: Background Removal.

(L) Spectrum with background; (R) Spectrum with background removed

methods are employed to characterize the background and subtract it from the pure spectrum.

Mechanical Filters: Mechanical filters are thin foils which are placed between the sample and the detector (or in the case of primary filters, between the primary beam X-rays and the sample). The X-ray absorption characteristics of the filter are unique to the foil material used. The filter will absorb or block some X-ray energies while allowing other energies to pass through. By selecting an appropriate filter material for a given application, peak overlap can be substantially eliminated.

Additional spectrum processing methods correct for artifacts in the spectrum resulting from specific, well characterized errors in the pulse processing electronics. These include peak shift correction, escape peak removal, and sum peak removal.

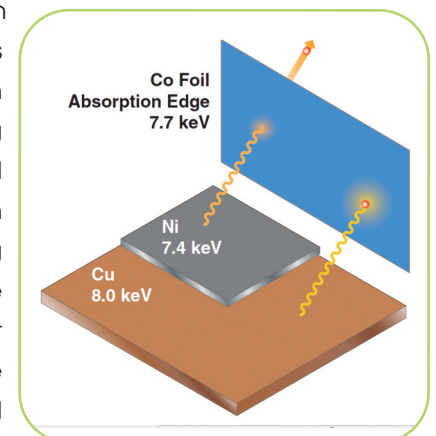


Figure 14: Mechanical Filtering

After spectrum processing methods are applied a net spectrum remains. By establishing an ROI within the net spectrum, net counts may be determined. For many applications, quantitative analysis may be enhanced by, or may require, the use of some or all of these spectrum processing techniques in order to perform accurate thickness/concentration calculations.

To convert processed spectral data into quantitative determinations requires mathematical models that relate ample X-ray count rates to thickness and composition. These models are called calibrations. An XRF tool can be calibrated using a variety of empirical and fundamental parameters techniques.

Empirical Calibration

By measuring the X-ray count rates from a coating/base combination of known thickness and/or composition (standard), we obtain the reference data needed to calculate the thickness and/or composition of unknown quantities of the same coating and base combinations. In practice, a series of standards with different values are measured to obtain the necessary reference count rates. Statistical methods are used to correlate the standard values to the measured XRF count rates.

Accurate calibration of the XRF tool is dependent on the availability of high-quality standards for the specific application under analysis.

Fundamental Parameters Calibration

The major practical drawbacks to empirical calibration methods are:

- The time required to measure a series of calibration standards.
- The difficulty of fabricating standard sets for intricate applications.

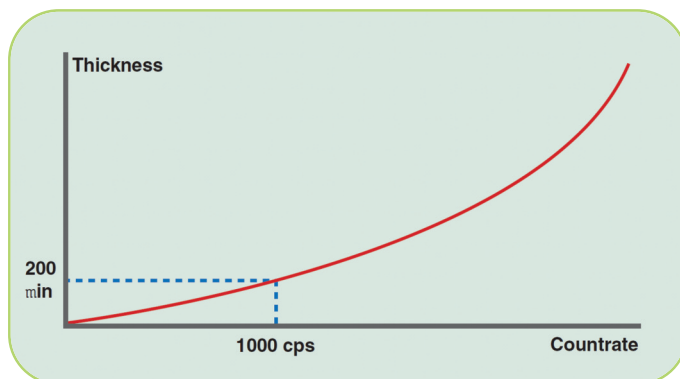


Figure 15: Calibration Curve

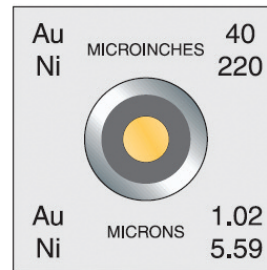
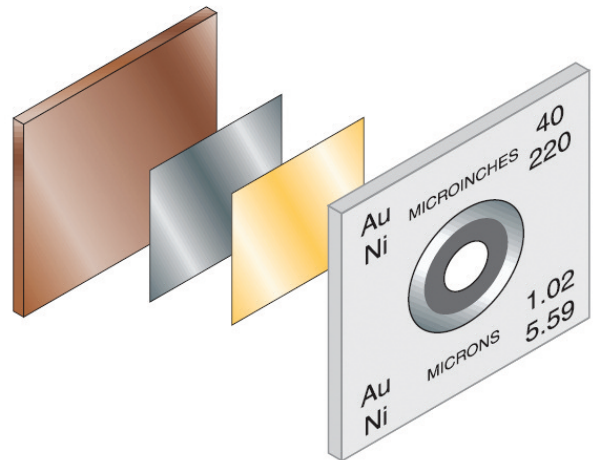


Figure 16: Typical Calibration Standard

Exploded view shows Gold (Au) and Nickel (Ni) foil interlayers on Copper (Cu) base layer.



Highly accurate mathematical techniques based on physical properties have been developed for a wide range of applications, enabling the XRF tool to be calibrated without (or with a reduced number of) standards.

In a fundamental parameters (FP) calibration, an extensive range of instrument parameters are first quantified. This information is incorporated into a mathematical model of the fundamental physics of X-ray interaction with the specific materials under analysis.

The mathematical model is often enhanced through the use of physical standards (the Standard-FP method). The improvement in test process throughput enabled by reducing or eliminating the need for physical standards can be substantial.

FP techniques offer versatility and often enhanced capability. For certain applications, however, empirical calibration offers a more accurate, simple, flexible, and reliable solution. An XRF tool should offer both calibration strategies.

Accuracy, repeatability, and reproducibility are crucial considerations when evaluating XRF tool performance. While related, it is useful to understand the distinctions between these terms.

Accuracy

Accuracy is an expression of the deviation of the mean measured value from the true value of the sample.

Assume, for example, that you have a sample with a coating thickness of 100 microinches. If, after a statistically significant set of measurements, you obtained a mean measurement value of 99 microinches, you could say that the system is accurate within one percent.

Measurement accuracy is primarily a function of calibration accuracy, which is in turn dependent upon the accuracy of the calibration algorithm, the quality of the standards used to calibrate, and the calibration measurement time.

Repeatability

The accuracy of any single measurement is highly dependent upon the precision, or repeatability, of the measurements. Repeatability is an indication of how well a series of measurements taken on the exact same spot of a sample yield the same or nearly the same readings. Repeatability is commonly expressed in terms of relative standard deviation (RSD) from a mean value.

Repeatability is highly dependent upon the total number of X-rays counted during measurement. X-ray counting is maximized by generating an incident beam of the highest intensity, using the largest beam size appropriate to the sample geometry, and by using the longest possible measurement duration.

Reproducibility

Reproducibility refers to an instrument's ability to obtain consistent measurement results when measuring the same sample at different times, with different operators, and possibly with different instruments of the same type.

Measurement System Gauge Capability R&R Study Worksheet													
Part/Asm Name	Disc			Gauge Name				Part No.	Side B				
Parameter	Thickness			Gauge No.				Measurement Unit	ul				
Tolerance (Spread)	50.00			Gauge Type	XRF			Zero Equals	0				
Measurement Time	15 sec.			Beam Size	30 Mills								
Operator	Frank				Joe				Doug				
Sample #	1st Trial	2nd Trial	3rd Trial	Range	1st Trial	2nd Trial	3rd Trial	Range	1st Trial	2nd Trial	3rd Trial	Range	
1	534.781	534.587	535.546	1.95960	534.840	536.126	536.506	1.28560	535.141	535.855	534.748	0.90750	
2	537.106	537.266	534.330	2.33950	538.175	533.864	536.649	4.51090	536.239	534.536	536.341	1.74520	
3	536.467	537.690	537.732	0.77680	537.762	535.703	537.823	2.11960	536.487	536.536	537.041	0.55420	
4	536.509	535.545	534.785	1.72470	534.531	535.117	533.968	1.14860	535.483	534.187	535.133	1.29550	
5	536.184	536.730	536.334	0.54640	537.700	536.755	536.906	0.94480	539.078	538.266	537.549	1.52890	
6	535.767	536.509	536.664	0.89700	536.482	536.649	535.490	1.16980	537.899	537.552	536.692	1.20660	
7	536.466	536.980	536.963	0.51460	536.875	537.277	538.283	1.40810	537.569	537.634	538.628	1.05890	
8	536.753	535.342	535.230	1.52340	535.502	537.864	536.534	2.36260	537.704	536.483	536.677	1.22110	
9	536.725	538.482	537.496	1.75730	537.944	536.956	537.628	0.98780	538.064	536.791	537.916	2.27220	
10	537.972	539.477	539.391	1.50540	538.186	539.424	538.487	1.23870	538.546	538.149	539.601	1.45200	
Average	536.673	536.861	536.607	1.35407	536.802	536.554	536.726	1.71769	537.321	536.585	537.033	1.32421	
Average-A	536.71363				Average-B	536.69382				Average-C	536.97942		
Test for Control:													
UCL-R = $D4 \cdot \bar{R}_{bar}$													
where $D4 = 3.27$ for 2 trials or 2.58 for 3 trials. Enter the appropriate $D4$ value:													
If any range value exceeds the UCL-R value, the measurement or reading should be reviewed or repeated, corrected, or discarded as appropriate, and the new average and ranges should be computed.													
Measurement System / Gauge Capability Analysis													
Equipment Variation (Repeatability) = $K1 \cdot \bar{R}_{bar}$													
where $K1 = 4.56$ for 2 trials or 3.05 for 3 trials. Enter the appropriate $K1$ value:													
Operator Variation (Reproducibility) = $K2 \cdot \bar{X}_{bar_diff}$													
where $K2 = 3.65$ for 2 and 2.70 for 3 operators. Enter the appropriate $K2$ value:													
Total Repeatability and Reproducibility Variation (R&R) =													
$(R\&R = \text{Square root of } (\text{Repeatability}^2 + \text{Reproducibility}^2))$													
4.53527													
%R&R = $100[(R\&R)/\text{Tolerance}]$													
9.07%													
Acceptability: An R&R of 10% or less is excellent; 11-20% is adequate; 21-30% is marginally acceptable; and more than 30% is unacceptable.													

Figure 14: Gauge R&R

The repeatability and reproducibility of any tool is commonly analyzed by the use of a Gauge R&R.

In the example at the left, three operators perform three sets of ten measurements each on samples using the same XRF tool. Overall sample measurement deviation must fall within specified tolerance (%R&R) to meet acceptable performance to be considered capable of controlling a process.

Precise and consistent X-ray beam to sample orientation is essential to ensuring accurate, repeatable, and reproducible XRF measurements. As sample structures become smaller, advanced optics and high-precision stage mechanisms for X-ray targeting are required to maintain overall XRF measurement performance.

Optics

A video image of the sample area is supplied by a video camera in the X-ray chamber and is displayed on the computer monitor (figure 18). Zoom optics are employed to adjust the field of view and magnification of sample surface detail. This aids in location and fine positioning of sample structures.

A computer-generated, auto-scaling reticle indicates the position and geometry of the X-ray beam at the sample. The operator adjusts the sample position to coincide with the X-ray beam spot using an input device such as a trackball or joystick, or by clicking directly on the video image with the mouse cursor. The system optics are calibrated so that when the sample in the viewing window is in fine focus, the required source-to-sample-to-detector distances are reproduced. Since X-ray intensities are a function of the inverse square law, maintaining these distances is crucial to obtaining reproducible measurements.

Advanced XRF systems utilize a directed laser beam to ensure precise, operator-independent focusing. By eliminating operator-dependent focusing, focal length variation is minimized, vastly improving overall reproducibility.

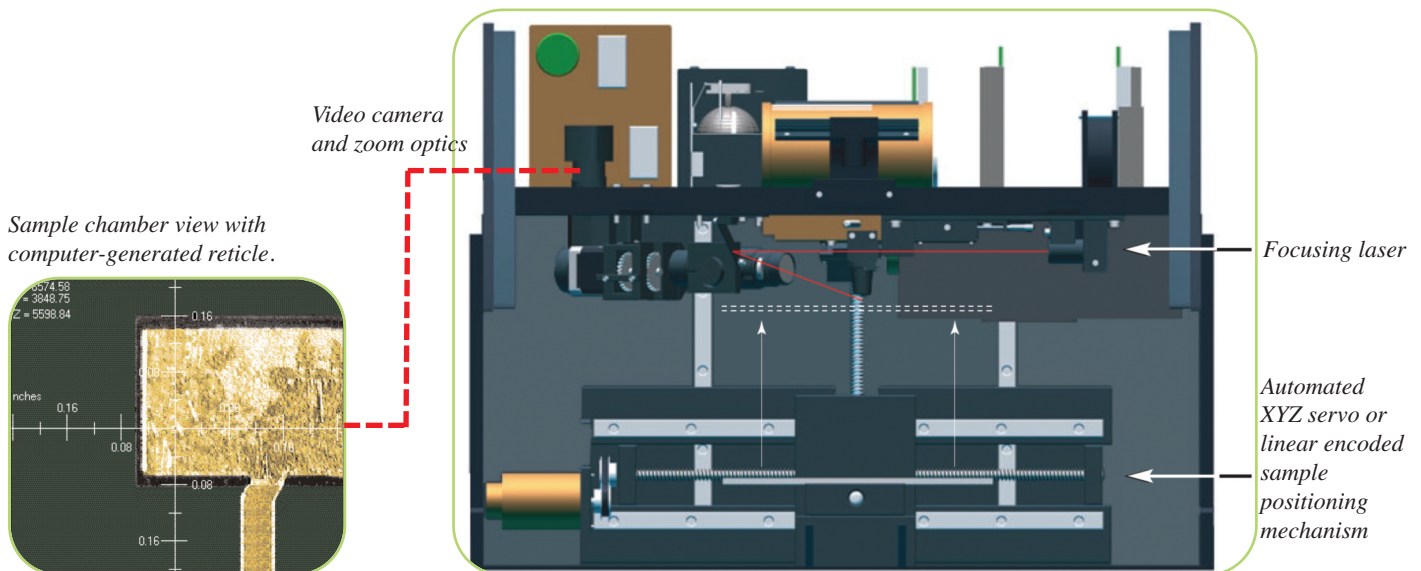
Stage

When measuring extremely small sample structures (in some instances so small that the incident X-ray beam and the area to be measured are close in size), a precision XYZ positioning mechanism is critical. In high throughput processes, inconsistent positioning will result in erroneous individual measurements.

High precision, servo-driven or linear-encoded stage drivers and the use of mechanical and optical positioning aids enhance positioning reproducibility and overall XRF tool throughput. When evaluating an XRF tool, the availability of the following features should be considered:

- Custom sample fixturing.
- Graphic motorized assisted sample positioning (“Point and Shoot”).
- Automated XYZ stage control recipes.
- Machine vision systems for local and global pattern recognition.

Figure 18: Stage and Optics



Software and interface design directly impact the return on investment realized from any process metrology tool. The XRF requires an interface that is easy to use, imposes minimal training requirements, and eliminates, as fully as possible, errors related to operator judgement.

Solar XRF tools feature a highly intuitive, graphically-driven operator interface running under Microsoft Windows™. Using simple point-and-click mouse commands, operators with little or no previous experience in XRF metrology can be testing within minutes.

Standard software features include:

Automated Stage Controls: Stored sample handling recipes ensure repeatable, reproducible X-ray beam targeting. Optional pattern recognition configuration enables instant, hands-free correction of sample positioning variations.

Statistical Tools: Including Mean, Standard Deviation, Percent Deviation, Pp/Ppk, and Min/Max, with Histogram, Trendline, X-Bar, and R-Chart display.

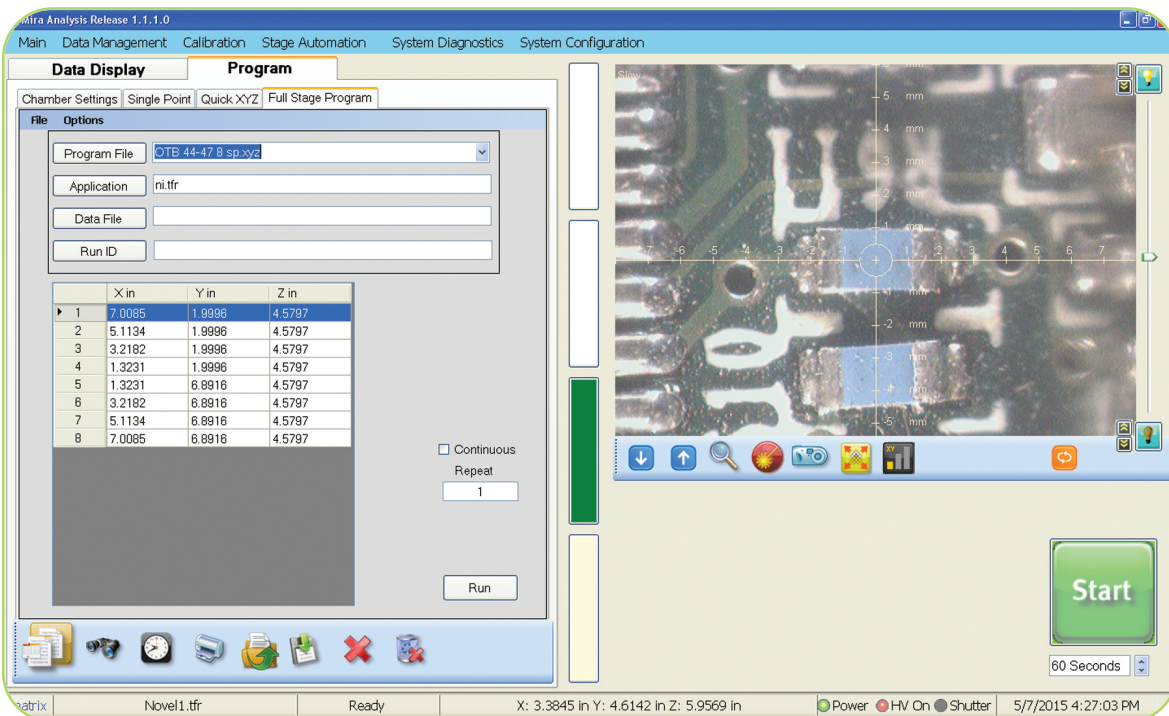
Visual Analysis Tools: Including three dimensional surface mapping/modeling; zoom optics and video image capture enable visual inspection of fine sample structures.

System Administration: Network compatibility; built-in security features for multi-user environments remote control software available.

Ergonomic Design: Integrated design of GUI, chamber, and input devices minimizes repetitive strain. Customizable interface streamlines measurement tasks.

Data Export: Measurement data can be exported and modeled using such popular programs such as Word, Excel, and Lotus.

Figure 19: System Software



Accuracy: Refers to how closely a measurement value obtained by an instrument conforms to the actual value of the sample.

Application: A specific combination of coating/base materials (or materials in solution) upon which XRF measurements are performed.

Background: Counts observed in the spectrum originating from X-ray scattering, electronic noise, and errors in pulse processing, rather than detected X-ray photons.

Baseline: Since characteristic energy data is derived from pulse height, a constant zero counts reference must be maintained. The XRF sets the baseline at the noise threshold level. Baseline restoration circuitry in the pulse processing electronics is designed to compensate for any baseline shift that occurs.

Bremstrahlung: Literally, "braking radiation". In XRF analysis, refers to the release of energy (in the form of X-ray photons) that occurs when electrons emitted by the X-ray tube filament change velocity and trajectory as they approach the nuclei of target atoms.

Calibration: Data, obtained by measuring reference standards or employing fundamental parameters, used by the XRF to create mathematical models for determining the thickness and/or composition of sample materials.

Collimator: A small aperture or optical focusing element used to shape and direct X-rays generated by the X-ray source.

Count Rate: The number of fluoresced X-rays per unit time counted from the sample under measurement.

Dead Time: Amount of time required by the XRF to detect a fluoresced X-ray and process the signal into a pulse. During this interval, other X-ray events cannot be detected or processed.

Detector: XRF component that produces output charges (pulses) that are proportional to the energy of X-ray photons entering the detector.

Escape Peak: A false peak in the spectrum produced by the occasional loss of some photon energy absorbed by the detector due to fluorescence induced in the detector medium.

Filter: A mechanical device (generally a foil) or mathematical technique used to distinguish X-rays fluoresced by materials with similar characteristic energy levels.

Fluorescence: The process by which incident electromagnetic radiation induces atomic ionization. As a result of ionization, electrons from higher energy orbitals drop (cascade) to lower energy orbitals. As a result of these transitions, characteristic energies are released by the atom in the form of X-ray photons.

Intensity: The number of X-rays counted by the detector at a given energy level or range of energy levels.

Multi-Channel Analyzer: Sorts detector output pulses according to energy level and counts the number of pulses accumulated at each level; from this information a spectrum (or pulse height analysis) is generated.

Peak: Channel in the spectrum containing the highest number of counts within a distribution of counts. The height and overall area of peaks within a spectrum yield quantitative information about the element(s) present within a sample material.

Peak Shift: Change in the position of peak(s) in the spectrum resulting from large changes in photon flux. Peak shift is an inherent characteristic of energy dispersive detectors.

Pulse: Analog output waveform produced by the XRF detector, electronics, and amplifier. Each pulse is proportional in magnitude to the energy of a detected X-ray photon.

Pulse Pile Up: Occurs when two X-rays enter the detector at nearly coincidental times. The pulse observed as a result will be approximately equal to the sum of the individual pulses, and is interpreted as originating from an X-ray with a much higher energy than either of the two incident X-rays.

Repeatability: The ability of an instrument to produce consistent results when performing multiple measurements on the same sample area. Also referred to as precision.

Reproducibility: The ability of an instrument to obtain consistent measurement results when measuring the same sample at different times and/or with different operators and/or using different instruments of the same type.

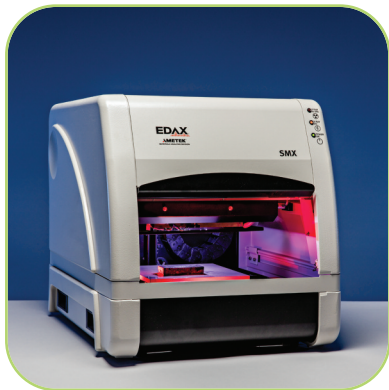
Spectrum: A frequency of occurrence histogram displaying the number of detected x-rays (counts) along its Y (vertical) axis, and their respective energy levels (in keV) along its X (horizontal) axis; used to make qualitative and quantitative determinations about sample materials.

Standard: A sample material of known thickness and/or composition used to calibrate the XRF unit for specific applications (see Calibration).

Sum Peak: Spectral artifact produced when two photons enter the detector simultaneously. A single pulse with an amplitude corresponding to the sum of the energies of the two photons is produced.

X-Ray: Radiation of extremely short wavelength produced by the bombardment of a substance by a stream of electrons moving at high velocity.

X-Ray Tube: An evacuated enclosure containing a filament and a quantity of target material. When high voltage is applied to the filament, electrons collide with the target material, inducing it to fluoresce X-ray photons.



SMX-BEN Coating Thickness Analyzer for development and QC of process control applications.



SMX-ILH Coating Thickness Analyzer for process control measurements at atmospheric pressure.



Orbis Micro-XRF Elemental Analyzer for the laboratory environment.

For more information about our products, please contact
info.edax@ametek.com

www.edax.com