

The Next Big Step in X-Ray FLUORESCENCE



TRIBOGENICS



A white paper on the technology behind the
Watson XRF metal analyzer featuring the world's first
field exchangeable X-ray cartridge

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The Next Big Step in X-Ray Fluorescence

X-ray fluorescence (XRF) spectrometry and analysis has undergone significant advancement over the past century making XRF the first choice for use in an increasing range of applications such as medicine, mineralogy, agriculture, manufacturing, construction, geology, and archeology. Among the most important advancements that are expanding the use of XRF are those that shrink the cost and size of the XRF instrumentation. The commercial release of the first hand-held XRF (HHXRF) technology nearly two decades ago enabled an important transition from stationary benchtop XRF instrumentation to mobile devices.

The commercial release of Tribogenics' triboluminescence technology in 2015 is the next big step in XRF technology because it nearly halves the cost of using XRF technology without sacrificing capability.

The first users that will benefit from this advancement are machining, manufacturing, and scrap recycling operators. It is also likely applications that have never considered using XRF technology before, because it was too expensive, may start benefitting from using it. This paper provides a brief overview of XRF technology. It explores how the technology works to enable you to more confidently decide how, where, and when your business can benefit from using this technology.

In 1895, Wilhelm Conrad Roentgen was studying cathode rays emitted from a high-voltage, gaseous-discharge tube. He noticed that a barium-platinocyanide screen, which just happened to be lying adjacent to the experiment, emitted a fluorescent light whenever the experimental tube was in operation even though it was encased in a cardboard box. This accidental and fortuitous discovery led to the identification of X-rays. In 1909, Charles Glover Barkla discovered a connection between X-rays radiating from a sample and the atomic weight of the sample.

Four years later, in 1913, Henry Gwyn Jeffreys Moseley discovered that there was a relationship between the atomic number of an element and the reciprocal of the wavelength for each spectral series of emission lines for each element. This provided the foundation for refining the periodic table by atomic number rather than by atomic weight and for making an X-ray spectrometer. However, it was not until 1948 when Herbert Friedman and Laverne Stanfield Birks, Jr. built the first XRF spectrometer, which opened the way for commercial use of XRF technology.

What is XRF spectrometry?

XRF is a phenomenon where a material emits photons as the result of being bombarded with enough high-energy X-rays. The types of atoms in the material and how they absorb the external x-ray energy affects the radiation energy (analogous to color for optical light) they emit. Each type of atom emits a different energy or color. An XRF spectrometer relies on this phenomenon to be able to identify what types and how much of each type of atom exist in a sampled material. By analyzing the energies or colors emitted by a material, it is possible to determine which elements are present. By analyzing the relative intensities of the energies or colors emitted by a material, it is possible to determine how much of each element is present.

XRF spectrometry is a non-destructive, non-intrusive analytical technique that can be used to determine the chemical composition of materials such as metals, glass, and ceramics in various states including solids, powders, and fluids. It is easier to use than other analysis techniques because many samples can be analyzed with little to no preparation and modern instruments are automated to operate under computer control, including set-up, calibration, taking measurements, and calculating results. XRF spectrometry also enables material identification to be determined in seconds to minutes depending on

the precision of analysis needed of the sample.

This combination of features makes XRF spectrometry practical and advantageous for many uses, including positive material identification (PMI), hazardous material detection, and certification verification for applications such as salvage, recycling, machining, and forensic science. XRF spectrometers are available in sizes ranging from cabinets, bench top units, and portable hand-held units.

The number and variety of applications that XRF can support expands each time the cost of using XRF instruments drops. So how are XRF instrument manufacturers able to lower the price of these devices?

Anatomy of conventional XRF spectrometry

There are a number of ways to implement XRF spectrometry, so to avoid confusion; this paper focuses on the specifics of XRF spectrometry using an energy dispersive XRF (EDXRF) approach. XRF spectrometers consist of four major sub-systems that form a signal chain: X-Ray tube, X-ray detector, multi-channel analyzer, and computer (see Figure 1).

The X-ray tube is where the X-rays are generated and directed at the target sample. The X-ray tube will house one of two types of X-ray

sources: radioactive or high-voltage sources. Radioactive sources are simple, small, and inexpensive; however, they cannot be turned off and pose sufficient environmental risks to the user and the community that there are registration requirements, restrictions on transportation and disposal, and periodic testing required to use radioactive sources. On the other hand, because high-voltage X-ray sources do not contain radioactive sources and they can be “turned-off” they do not suffer from the same limitations. However, they do require a source of high-voltage electricity to be able to generate and emit the desired X-rays.

The high-voltage supply is combined with a target anode, which typically comprises of atoms of a single element, to generate the desired X-rays. The high-voltage supply is generated by transforming a low-voltage battery source to the needed voltage (20kV to 100kV). The high voltage supply is connected to the X-ray tube via a high-voltage connection or a mono-block structure (see Figure 2).

The X-ray tube contains a vacuum housing with a wire filament and a target anode inside. An electric current applied to the filament heats it up to about 1000 degree Celsius so that it emits electrons. Once the filament is emitting electrons, the high-voltage is applied across the filament and the target anode, which accelerates the electrons from the filament towards the target anode. The interaction between the accelerated electrons and the target anode causes the emission of X-

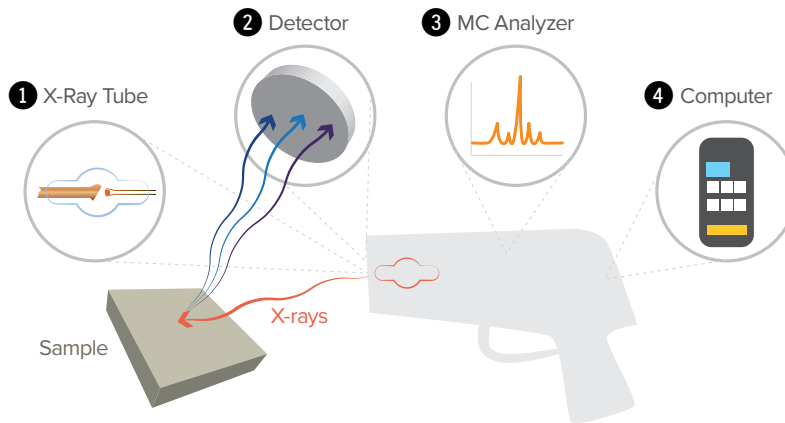
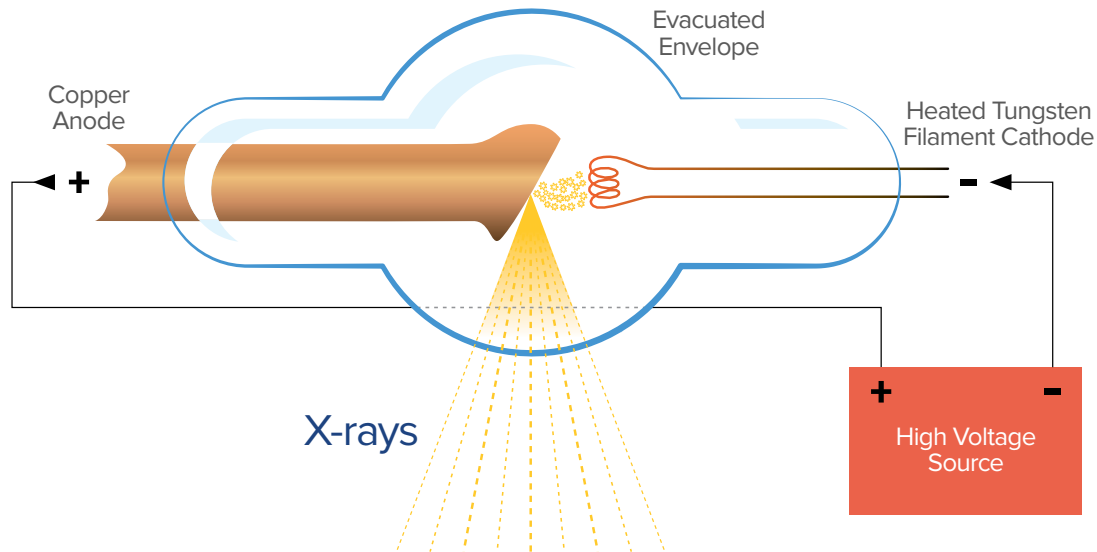


Figure 1: XRF spectrometry relies on four major sub-systems that form a signal chain: X-ray tube, Detector, Analyzer, and computer.

Figure 2: Contemporary X-ray tube with complex high-voltage power supply, filament, anode, etc.



rays. The type of element in the target anode determines the energy of the emitted X-rays.

The X-ray detector is used to measure the fluorescent X-rays emitted from the target sample. There are different types of detectors available for XRF applications. EDXRF systems typically use solid-state detectors, such as a Si-PIN detector or a Silicon Drift Detector (SDD). Each type of sensor has advantages in different applications; neither is always best. The resolution and sensitivity are two important properties of these detectors. A higher resolution means the detector can detect the difference between more energy levels. A higher sensitivity means that a higher percentage of incoming photons are detected.

The Si-PIN detector has the advantage where high detection sensitivity is important but resolution is not so critical. The Si-PIN detector also shines where cost is important because the SDD is more complicated to manufacture so it is more expensive than a Si-PIN. On the other hand, the SDD is a newer detector technology that has better energy resolution and supports higher count rates than a Si-PIN with the same area – so it is better for applications where the highest resolution or support at high count rates is needed.

These types of solid-state detectors are able to capture X-ray photons that pass through their detector window and produces electron-hole

pairs based on the number of photons captured. The higher the energy of each photon, the more electrons it will produce. A high voltage across the front and back of the detector attracts the electrons to the back. The arriving electrons cause a potential drop and generate a negative pulse, which is proportional to the number of electrons and by extension proportional to the incoming X-ray energy. The multi-channel analyzer receives and counts the pulses from the X-ray detector. It creates a histogram of the energy detected from counting the pulses from the X-ray detector. This enables the analyzer to differentiate between and identify the radiation signature from the various elements in the target sample.

The computer manages the user interface, communications, as well as data storage, retrieval, and display. XRF spectrometers were able to become mobile, hand-held devices, in part, because the computer functionality was able to reside on smaller embedded application processors that supported the small form factor. The computer interface and value-added analytical functions it supports represents an important area of differentiation between similar XRF devices. It is typical for the computer to use a common operating system with touch screen support, including Microsoft Windows or Android, so as to make it simpler for people to use these tools. Communication options continue to mature enabling users to safely and securely store

and retrieve thousands of results over wired and wireless interfaces.

As much as the computer might be the expected sub-system for new XRF innovations, Tribogenics has developed an important innovation in the X-ray tube portion of the system that relies on triboluminescence.

Triboluminescence – Enabling major advances in XRF

Triboluminescence is the phenomenon of creating light through mechanical action such as pulling apart, ripping, scratching, crushing, or rubbing different materials. For example, this phenomenon is observable when breaking sugar crystals and peeling adhesive tapes. This phenomenon has been known since ancient civilizations, but in the 1980s, a team in Russia reported that crushing certain types of rocks in a vacuum produces luminescence in the X-ray energy range. In 2008, a team of physicists at UCLA backed by DARPA funding expanded on this discovery and confirmed that they could use triboluminescence to generate X-rays in a useful and repeatable way.

It turns out that using triboluminescence to generate X-rays can have a profound impact on lowering the complexity and cost of the X-ray tube. It is now possible to rely on the triboelectric effect caused by mechanically pulling materials together and apart to discharge enough electrons at the target anode to generate the necessary amount of X-rays to successfully perform XRF spectrometry. In short, a mechanical system replaces and eliminates the need for a high-voltage supply to generate X-rays. This is the primary innovation that is driving down the cost of entry for XRF spectrometers by almost half and creating many new uses for HHXRF.

Simplifying X-ray generation

The high-voltage power supply and supporting components are no longer needed. All of the other sub-systems in the XRF spectrometer remain the same. An electric motor, battery, switch, microcontroller, and a low-voltage connector replace the need for inverters, transformers, and control system needed for the high-voltage supply (see Figure 3). There is no thermal cycling because there is no need to heat up a filament. It is no longer necessary to provide a cable or monoblock connection between the high-voltage supply and the X-ray source.

Eliminating the high-voltage power supply affects how a user maintains the XRF spectrometer. It is now safe for a user to change the X-ray tube without needing extensive safety training and certification because there are no high-voltage interfaces to be careful of when opening the case of the XRF spectrometer. It is no longer necessary to send the HHXRF unit to the shop for routine X-ray tube replacement or calibration because users are able to replace the cartridge containing the X-ray tube in the field themselves.



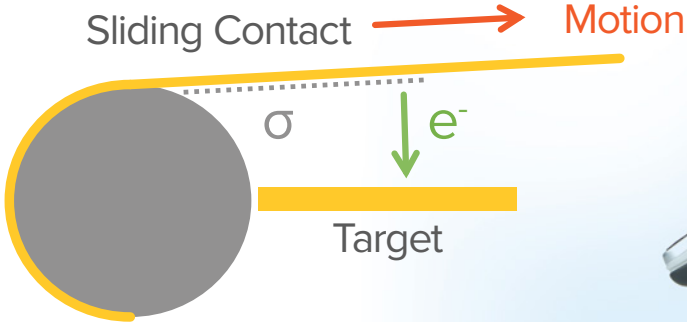
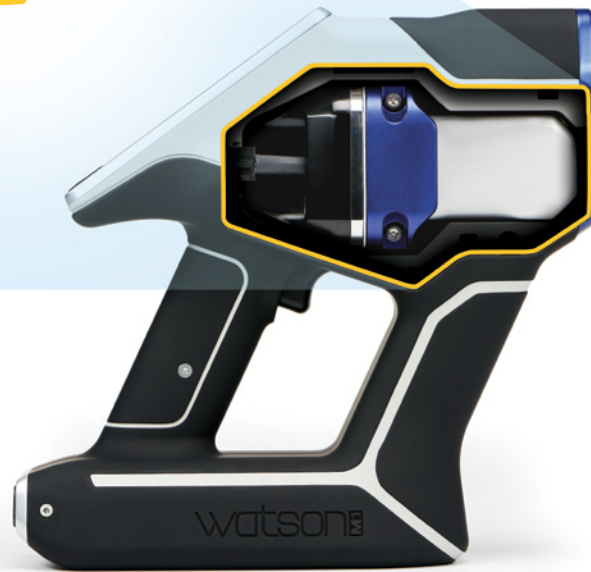


Figure 3: X-ray tube using triboluminescence creates a simpler, less costly XRF spectrometer.



New applications

At approximately \$25,000 to \$30,000, XRF spectrometers have come a long way from the \$250,000 benchtop units. Now, with the introduction of Tribogenics' \$10,000 HHXRF spectrometers, the opportunity for XRF to be used in new functions and application spaces is inevitable. Similar to how dropping prices for personal computers migrated computers from being shared resources between a few people to dedicated resources for each person, this new entry level cost threshold expands the contexts for using XRF spectrometers.

The lower cost threshold opens the door for richer differentiation at the lower end of XRF spectrometers in the form of pure lower prices, added integrated premium capabilities, or a combination of both in new devices. Pure lower priced devices might act as hot spares or remote, mobile alternatives for more expensive units.

The lower cost threshold provides room in the design budget to improve the user interface and analysis components as well as add robust, wireless, and cloud-based communication capabilities currently not available.

Because XRF is a non-invasive, non-destructive process, the lower cost (less than \$1/measurement) more easily permits multiple measurements throughout the lifecycle of a material or component to perform live verification of the material you are working with. Low-cost, mobile XRF spectrometers complement rather than replace site spectrometers and lab testing. Personnel will be able to test critical components before and after they are put into service.

Coupling the XRF instrumentation with wireless communication makes it possible for higher-skilled personnel in the central office to more directly assist lower-skilled personnel in the field resulting in the ability to deploy more capabilities in the field without incurring the cost for high-skill requirements everywhere.

The future of XRF spectrometry is bright. Ask us to show you how to decide if it is the right time to add XRF to your business. Learn what new ways XRF spectrometry is being used.

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