The Selection of an X-ray Source Target Anode Material for Hand-Held X-ray Fluorescence Instruments

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The Selection of a Target Anode Material for XRF

The basic premise of XRF is that high energy x-rays are aimed at a sample and then the atoms in the sample may absorb an x-ray. This ionizes the atom by displacing an electron from one of the shells close to the atom. The next step is for an electron from an outer shell to fall into the shell vacancy, emitting the excess energy in the form of characteristic x-ray radiation. The energy of the x-ray is dependent on the energy levels of the shells participating in the process and therefore is "characteristic" to the material of the sample.

A critical detail for the x-ray source used for XRF is that x-ray source needs to produce x-rays at energies higher than absorption edge of the element in order to ionize the atom. Every element has different absorption edges, just as they have different characteristic x-ray lines. The x-ray source needs to be tuned in various ways for effective detection of different elements.

There are three basic methods for tuning the spectrum-shape from the x-ray source:

- Setting the high voltage on the x-ray tube.
- Putting an x-ray filter between the x-ray source and the sample.
- Selection of the anode material on the x-ray tube.

This report will focus on the selection of the anode material on the x-ray tube. For additional information about setting the high voltage or selecting a filter please go to <u>www.moxtek.com</u> and select the "Consult our Experts" option in the bottom right of the page.

X-ray Anode Selection

Unlike the high voltage or the filter, the anode of the x-ray tube is fixed; you are unable to change an anode material due to this material being on the inside the vacuum of the x-ray tube. Getting the anode material correct is critical for both a single XRF application instrument, as well as instruments for multiple XRF applications, because, in many ways, it defines the application capabilities of the instrument. Therefore, the choice of anode material strongly depends on the particular XRF application.

For HHXRF, there are two major classes of x-ray tube anode types, 'light element' anodes, specifically for exciting light elements with absorption edges below about 3 keV, and 'general

use' anodes for exciting elements higher than about 3 keV. Each class of anode will be discussed in the two subsections below.

Anodes for Light Element XRF Detection

In handheld XRF, light elements are defined as being from Mg (Z=12) to Cl (Z=17) in the periodic table. Light element XRF detection is a very technically demanding application, especially for HHXRF, and it determines most of the design limitations on x-ray sources for HHXRF, as well as on the x-ray detectors.

X-ray tube anodes for light element detection in HHXRF are most commonly made of either rhodium or silver. These elements are ideal for light element excitation in a sample because they both have L lines which are near 3 keV: silver has L lines between 2.9 and 3.4 keV and rhodium has L lines between 2.7 and 3.0 keV. The L line strongly excites fluorescent lines just under 3 keV, such as from S, P, Si, Al, and Mg, which are the specific elements of interest for light element XRF. HHXRF x-ray sources designed for light element analysis use a very thin (~125 μ m, or thinner) beryllium window on the x-ray tube. This thinner window is for the purpose of minimizing x-ray absorption of the Rh or Ag L lines in the x-ray window itself. In these light element applications there is no x-ray filter. Air is often an unwanted filter, absorbing roughly 25% of the x-rays between 2.5 and 3 kV in about 1 cm of air. (For reference, a 3kV x-ray has a wavelength of about 0.4 nm).

Often, x-ray sources used for light element detection are used for other XRF applications as well, which require running the x-ray source at a high voltage of 40 to 50 kV. This presents a design constraint in that an x-ray source: a source optimized for 10-20 kV operation is not optimized for 40-50 kV. For HHXRF almost all the tubes are a transmission anode design, which consists of the target material deposited directly on the x-ray window with a thickness of about 1 µm. The anode thickness is optimally set to be thick enough to stop the x-ray tube's electron beam, and then thin enough to not cause any additional filtering of the generated x-rays. In the design of the anode thickness, this optimum is specific for a single high voltage (Figure 1).



Figure 1. This figure shows the electron interaction of a 1 um W foil with a 10, 20, and 30 kV electron beam. This thickness will have an optimal performance at 20 kV. Below 20 kV, the extra thickness of the foil acts as an x-ray filter, and above 20 kV, electrons get through the tungsten target into the beryllium window and do not generate the wanted x-rays.

For light element detection, getting a large L line from the x-ray source onto the sample to excite the light elements is paramount. Yet the same x-ray source is needed for XRF applications with higher voltages as well. This leads to a compromise in the design between the two applications.

The rhodium or silver anode layer has a few target thickness options for operation between 20 to 40 kV. This range of target thicknesses is split between the two application needs, for light element XRF and other XRF applications. An x-ray tube manufacturer will have a selection of proprietary thicknesses optimized over this range for HHXRF, with the exact target thickness picked by the instrument manufacturer (Tables I and II). Figure 2 shows the x-ray flux advantage and disadvantage of three different rhodium anode thicknesses at different voltage settings. Since the light element XRF detection is so much more challenging, any HHXRF instrument made for specifically for light element detection will almost always use the thinner rhodium target, RH3. The RH3 target thickness increases the L line x-ray signal below 20 kV, which is exactly the reason that this thickness is most often chosen. The x-ray flux drop off for settings 30 to 50 kV is not wanted, but applications using the higher voltages still work reasonably well, even with the large reduction in x-ray intensity.

Target	Target	Optimized		a	a	a	a	a
ID	Material	kV		10 kV	20 kV	30 kV	40 kV	50 kV
RH3	Rh	20.0	e- anode Sim.	0.95	1.00	0.87	0.62	0.38
			X-ray L-lines	0.58	1.00	0.81	0.53	0.36
RH2	Rh	25.0	e- anode Sim.	0.90	0.99	0.99	0.90	0.70
			X-ray L-lines	0.45	0.84	1.00	0.82	0.60
RH7	Rh	h 30.0	e- anode Sim.	0.87	0.99	1.00	0.96	0.82
			X-ray L-lines	0.43	0.77	1.00	0.85	0.63

Table I. This table gives the calculated and measured x-ray flux output of three Rh anode thicknesses produced at Moxtek, optimized for 20 kV (RH3), 25 kV (RH2), and 30 kV (RH7). For each anode thickness, the line labeled 'e⁻ anode Sim' shows the calculated emission of the X-ray L lines from the anode. For each anode thickness, the line labeled 'X-ray L-lines' shows the experimentally measured x-ray intensity in the L lines, normalized to the x-ray tube power. The electron modeling trends line up well with the experimentally measured x-ray intensity. The differences between the two are due to the modeling not taking everything into effect such as self anode absorption and x-ray window absorption.

All the arguments for the rhodium anode apply equally well to the silver anode. The choice between the two target types is often to the decision of a particular instrument manufacturer. A downside to the rhodium target is the rhodium line at 2.70 keV is very close to the K line for Cl at 2.62 keV. Several of the Rh L energies do not excite Cl, and the peaks are close together causing peak overlap with Cl. In this particular case of Cl detection, a silver anode is a better choice.



Figure 2. The figure on the left shows the flux intensity for the Rh L lines as a function of the voltage on an x-ray source, normalized to the x-ray tube power of 1 Watt. For light element detection, the target of choice is RH3, a thinner anode, which gives roughly a 20% increase in the L line intensity at settings between 5-20 keV. The disadvantage of the RH3 anode thickness is that there is a loss in overall x-ray intensity of roughly 40% at settings between 30 to 50 kV at the high end, which most HHXRF manufacturers find acceptable.

Target ID	Target Material	Optimized kV		@ 10 kV	@ 20 kV	@ 30 kV	@ 40 kV	@ 50 kV
AG2	Ag	20.0	e- anode Sim.	0.94	1.00	0.91	0.70	0.45
			X-ray L-lines	0.56	1.00	0.82	0.53	0.36
AG1	Ag	40.0	e- anode Sim.	0.83	0.98	0.97	1.00	0.93
			X-ray L-lines	0.36	0.70	0.95	1.00	0.83

Table II. This table gives the calculated and measured x-ray flux output of two Ag anode thicknesses produced at Moxtek, optimized for 20 kV (AG2) and 40 kV (AG1). For each anode thickness, the line labeled 'e anode Sim' shows the calculated emission of the X-ray L lines from the anode. For each anode thickness, the line labeled 'X-ray L-lines' shows the experimentally measured x-ray intensity in the L lines, normalized to the x-ray tube power. The electron modeling trends line up well with the experimentally measured x-ray intensity. The differences between the two are due to the modeling not taking everything into effect such as self anode absorption and x-ray window absorption.

Anodes for Other XRF Applications

General use anodes for HHXRF are most commonly made of tungsten, and work with many XRF applications, with light element XRF being a notable exception. Tungsten anodes produce more flux per unit current, the reason being that the bremsstrahlung radiation production scales with the atomic number 'Z' of the target. (Handbook of Optics, Third Edition Volume V (2009) Chapter 54) Additionally tungsten is a very robust material, being a refractory metal with a high melting point, which often gives it the edge over other materials. Sources that have a tungsten anode have a beryllium window thickness of 250 µm or more. Tungsten anodes are almost never used for light element XRF, so a thinner window is of no value. The tungsten anode thickness is optimized for the highest kV the source typically produces. For example an HHXRF instrument for scrap metal sorting has an x-ray source fixed at 35 kV. The tungsten anode thickness on the transmission anode source would likewise be optimized for 35 kV (Figure 3). Table III gives a list of typical tungsten anode thickness as well as the kV the anode is optimized for.

Target	Target	Optimized		@ 10	@ 20	@ 30	@ 40	@ 50	@ 60	@ 70
ID	Material	kV		kV						
W01	W	25.0	e- anode Sim.	0.88	0.97	0.96	0.82	0.58	0.39	0.26
			X-ray 3.5-10 keV	0.25	0.75	1.00	0.92	0.72		
W07	W	40.0	e- anode Sim.	0.77	0.87	0.98	0.99	0.95	0.78	0.58
			X-ray 3.5-10 keV	0.15	0.54	0.84	1.00	0.96		
W06	W	60.0	e- anode Sim.	0.37	0.69	0.90	0.96	0.98	1.00	0.94
			X-ray 3.5-10 keV	0.01	0.32	0.52	0.68	0.81	0.94	1.00
W-R	W		e- anode Sim.							
			X-ray 3.5-10 keV	0.37	0.79	0.95	1.00	0.83		

Table III. This table gives the calculated and measured x-ray flux output of three W anode thicknesses produced at Moxtek, optimized for 25 kV (W01), 40 kV (W07), and 60 kV (W06). W-R is an thick refection anode, like more traditional x-ray tube designs. For each anode thickness, the line labeled '*e* anode Sim' shows the calculated emission of the X-ray L lines from the anode. For each anode thickness, the line labeled '*X-ray L-lines*' shows the experimentally measured x-ray intensity between 3.5 to 10 kV, normalized to the x-ray tube power. The electron modeling trends line up well with the experimentally measured x-ray intensity. The differences between the two are due to the modeling not taking everything into effect such as self anode absorption and x-ray window absorption.

XRF applications that use a tungsten anode are very often used in conjunction with a filter exterior to the tube. Two notable filters are an aluminum filter of about 0.5 mm thickness for scrap metal sorting, and a copper filter for hazardous element detection.



Figure 3. This figure shows the x-ray intensity of several different tungsten anode types optimized for different high voltages, normalized to the x-ray tube power of 1 Watt. This is the flux through a copper filter, which is often how such a source would be used.