X-ray Detectors

X-ray Fluorescence for Non-Aluminum Metals/Alloy Identification: SS304 Identified with an SDD, XPIN6 and XPIN13

Application Note

Moxtek Inc. Orem, UT, USA
S. Cornaby, Ph.D.

Introduction

The question being addressed is: "**What is the best detector to use for non-aluminum metal and alloy identification, an SDD or a PIN detector?**" This paper shows that both a PIN and a SDD detector are equally adequate for identifying many common non-aluminum metals and alloys. The results section will focus the explanation on stainless steel 304 as an example material. Therefore, for this application **a PIN detector will work perfectly well and will be preferable to an SDD given the price difference.**

Comparing Detectors

The two main types of energy dispersive detectors are PINs and SDDs. The table below outlines a brief comparison between the two:

	Detection Area	Fe^{55} Resolution	Detector Inner Temp.	Upper Input Count Rate (ICR) Limit	Price
Typical SDD	10 - 50mm^2	120 - 160eV	-20 to -40°C	~500kcps	More expensive
Typical PIN	5 - 15mm^2	150 - 220eV	-20 to -40°C	~100kcps	Less expensive

Table 1: A brief comparison between typical SDD and PIN detectors.

A typical SDD has better performance over a PIN; they have a better ultimate energy resolution, and they are able to count more x-rays in a given time. The resolution is important in resolving x-ray events from different elements, and the counting is important for getting better statistics in a shorter time frame. A typical PIN detector has the major advantage of being less expensive, and is often used in XRF systems that are price sensitive.

PIN detectors are a good fit for many XRF applications, such as metal and alloy identification, which do not need the performance advantages that the SDD provide.

Experimental Conditions

The testing has a standard XRF set-up, shown in Figure 1. Moxtek's 50kV, 4 Watt Ultra-Lite x-ray source with a tungsten anode and a 250µm beryllium window. The source was set at 50kV and at 15 or 20µA. The source-to-sample distance is 25mm, with a 70µm copper filter in front of the source. The sample-to-detector distance is 25mm for each detector. Both XPIN detectors have a 25µm thick beryllium window and SDD has a 12µm beryllium window. The signal from the detector was processed by Moxtek's MXDPP-50. The x-ray source and the detectors have an aluminum sleeved brass collimator on them. The collimator is 11mm long with a diameter of 3.8mm. The aluminum sleeve is necessary to eliminate the stray XRF signal from the brass, insuring the XRF signal is coming exclusively from the sample.

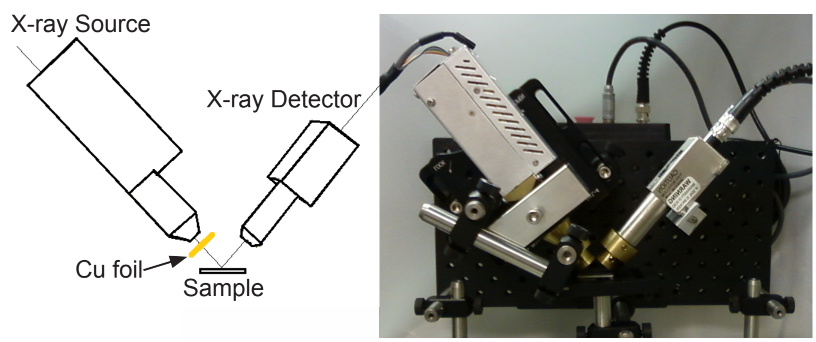

Figure 1. On the left is a sketch of the XRF setup, outlining the most critical parts. On the right is an image of the set up where all the components including collimators can be seen.

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The Cu filter eliminates most of the x-rays from the source below ~15keV, giving a better signal-to-noise ratio in this region, but the Cu filter does let one tungsten L_α line through at ~8.3keV. The tungsten L_α line improves the excitation from Nickel and the lower Z elements, but also leads to a non-XRF peak which may confuse the untrained operator or an XRF algorithm. Figure 2 shows the XRF spectra from a clean plastic sample, comprising the Compton scattered tungsten L_α line and Compton scattered bremsstrahlung from the source. An SDD, XPIN6 and XPIN13 detector where compared for their XRF performance in identifying non-aluminum alloys and metals. Table 2 outlines the critical technical merits of each detector in this experiment.

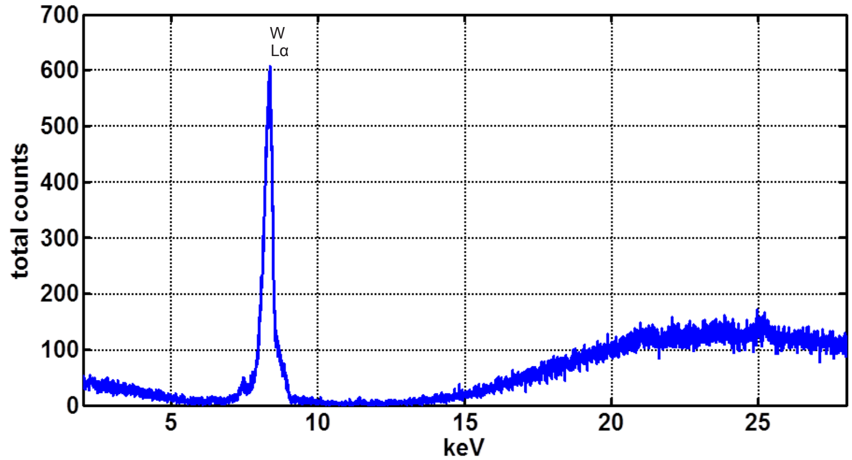

Figure 2. Spectra collected from a plastic sample, which shows the Compton scattered background from a clean XRF sample.

	Detector Area	Detector Thickness	Fe^{55} FWHM resolution	SS304 Spectra counts in 30 sec	Dead time	DPP Peaking time	Tube current	Detector Temp
SDD	20mm²	500μm	150eV	349k	26%	8μsec	20μA	-45 °C
XPIN6	6mm²	625μm	165eV	117k	22%	20μsec	20μA	-35 °C
XPIN13	13mm²	625μm	200eV	131k	25%	20μsec	15μA	-35 °C

Table 2. A functional comparison between an SDD, XPIN6 and XPIN13 detector. Each detector was tested in a XRF setup (Figure 1) for non-aluminum metal alloy detection.

For a quazi-normalized XRF performance comparison, each detector was set to run at about a 30% dead time by adjusting the tube emission current. The SDD, as expected, has higher technical performance than the PIN detectors. The SDD has a lower Fe^{55} FWHM resolution at a faster peaking time and more detecting area. This results in the SDD having roughly a 3X higher counting rate than the PIN detectors.

XRF Results

Stainless steel 304 is comprised of <0.03% C, <1% Si, <0.045% P, <0.03% S, 17.5-20% Cr, 8-11% Ni, and the balance in Fe. This XRF setup is not adequate for detecting elements below calcium on the periodic table, therefore only the elements of chromium and above will be detected in the SS304.

Each detector recorded an XRF spectrum from a 304 stainless steel source for 30 seconds. Figure 3 shows the full spectrum from the SDD, XPIN6, and XPIN13, showing all the major elements. Figure 4 shows the same spectral data focused on the region from 5 to 9keV. One key point is all the element's K_α lines are separated well enough for clear identification.

The collected spectra from the stainless steel 304 sample was run through an XRF fundamental parameters (FP) routine to turn the spectra into elemental concentrations. FP uses first principles; one inputs the tube settings, the detector characteristics and the spectrum; the algorithm then outputs elemental concentrations. Table 3 below outlines the physical parameters in the XRF set-up, which are needed as inputs into the FP routine.

Each detector, when properly set up in the FP program, gives nearly the same results. Table 4 below outlines the resulting concentrations from each detector. 30 second and 10 second XRF scans were taken on each detector. For comparison to the industry, scans of about 10 seconds or less is usual for handheld XRF instruments for metal identification. Comparing the 30 to 10 second scans show that the identification can easily be achieved for all the detectors in 10 seconds. The higher count rates of the SDD are not explicitly needed for sub-percent level element identification in short time frames of about 10 seconds for alloy and metal identification.

Using the FP routine, each detector gave an elemental concentration within 1% or less for each of the elements compared to each other detector. This level of accuracy is adequate for identifying stainless steel 304.

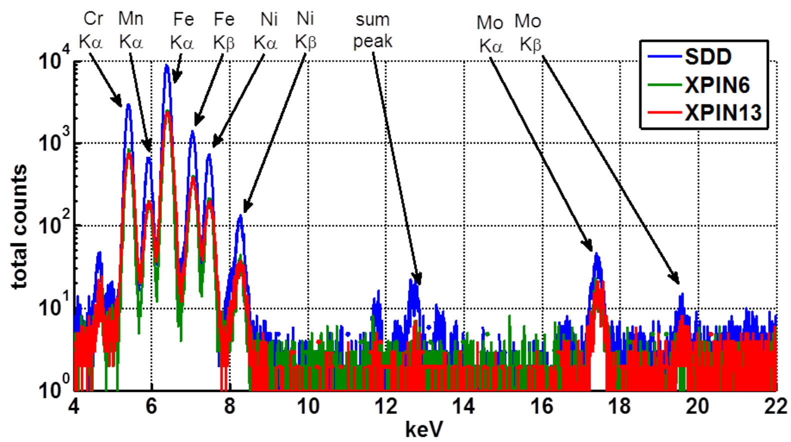

Figure 3. XRF Spectra collected from a 304 stainless sample, over 30 seconds, with all the major peaks labeled. Y axis of counts is in a log scale.

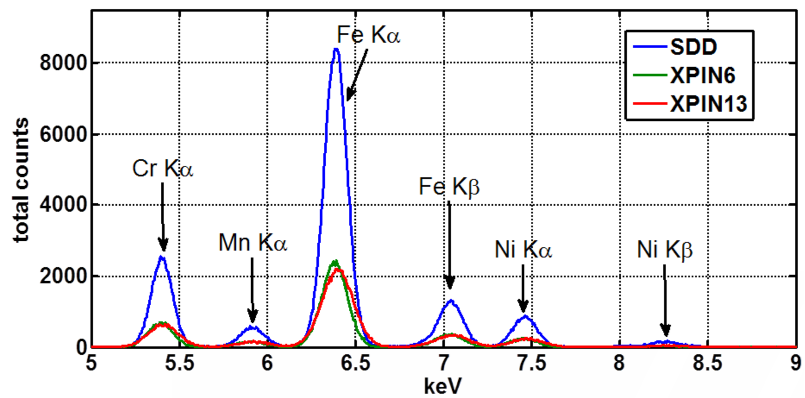

Figure 4. The same XRF Spectra collected in Figure 3, with a linear y-scale and energy range from 5 to 9keV.

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Tube set-up	Tube Target	High Voltage	Beryllium Window Thickness	Tube- to-sample	Inc. angle/ take off angle	X-ray Source Filter
	W	49.3kV	250µm	25mm	90° / 90°	Cu, 75µm
Detector set-up	**Detector Active Thickness**	**Detector Dead Layer**	**Beryllium Window Thickness**	**Sample- to-detector distance**	**Inc. angle/ Emer. angle**	**Detector Filter**
SDD	500µm	0.15µm	12µm	25	135° / 45°	none
XPIN6	625µm	0.15µm	25µm	25	135° / 45°	none
XPIN13	625µm	0.15µm	25µm	25	135° / 45°	none

Table 3. The experiential inputs needed by the FP program to calculate the elemental concentration. The region of interest (ROI) of the FP program ranged from 2 to 40keV.

	Cr	Mn	Fe	Ni	Mo	Co	Cu		Total counts in spectra
Tabulated SS316	**17.5-20%**	**<2%**	**b**	**8-11%**	**---**	**---**	**---**		
SDD-30 sec	18.3	1.5	71.8	7.7	0.09	0.03	0.59		349k
PIN6-30 sec	18.3	1.7	71.5	8.0	0.12	0.03	0.31		117k
PIN13-30 sec	18.5	1.7	71.3	8.1	0.12	0.02	0.16		131k
SDD-10 sec	18.5	1.7	71.0	7.9	0.09	0.03	0.58		117k
PIN6-10 sec	18.5	1.4	71.5	7.8	0.11	0.02	0.08		51k
PIN13-10 sec	18.4	1.4	71.7	7.6	0.13	0.03	0.64		61k

Table 4. The resulting concentrations from the three compared detectors using the 30 second and 10 second scans at ~30% dead time from a SS304 sample. The last column gives the total number of x-ray events collected in each of the spectra.

Conclusion

Both a PIN and a SDD detector are equally adequate for identifying many common non-aluminum metals and alloys, such as stainless steel 304. Therefore **a PIN detector will work perfectly well and will be preferable to an SDD given the price difference for this application.**

MOXTEK®
452 West 1260 North / Orem, UT 84057
Phone 801.225.0930 / Fax 801.221.1121
www.moxtek.com
info@moxtek.com