

## Application Note

### ComPact 5/PIN



## Use of Semiconductor Detectors (PIN – Diodes)

### Introduction

For more than 20 years, dedicated X-ray coating thickness analysers have been on the market. Typical for these types of instruments is a low power, air cooled X-ray tube, a small excitation beam size ( $< 500\mu\text{m}$ ), and a small footprint (table top). To achieve this small beam size in a very cost effective way the primary beam is collimated by an aperture defining the size. This means that most of the primary X-rays are blocked. To compensate for this, detectors with a large active area have been used, typically gas filled proportional counters. Unfortunately these detectors have a relatively bad energy resolution. The most common type of detector used in coating thickness analysers has a Xenon gas filling, an active area larger than  $1000\text{mm}^2$ , and a Be-entrance window. The energy resolution is about 1000 eV FWHM for Mn K $\alpha$  and about 1750 eV for Ag K $\alpha$  X-rays. Especially with more complex alloy multilayer coatings this can be a real disadvantage. Even though 20 years ago semiconductor detectors, so called Si(Li) – detectors, with excellent energy resolution have been available their use is not very practical because they require cooling with liquid Nitrogen, and in a normal production area this is not very practical.

### New Peltier- cooled Si-PIN-diodes

Si-PIN-diode detectors, which have been available for a few years, are made out of very pure silicon, so-called intrinsic material. This allows the creation of a detector that does not need to be cooled with LN. A simple, electrical Peltier-cooler is good enough. In the beginning the size of PIN-diodes were very limited, a few  $\text{mm}^2$  only. Further, the active thickness of the diode was 0,3 mm only. Today, PIN- diodes with  $25\text{mm}^2$  and 0,5 mm thickness are available. In particular for higher energies the increase in thickness has a positive effect. According

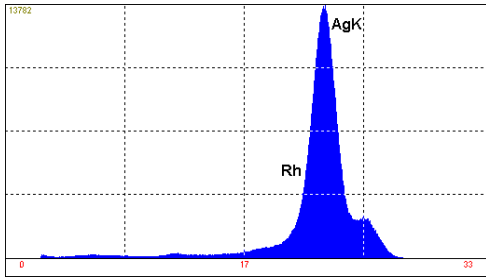
to manufacturers data for Ag K $\alpha$  X-rays a 0,3 mm thick PIN- diode has an absorption efficiency of 18% while a 0,5 mm thick diode has about 30%. ( For comparison, a Xenon filled gas proportional counter has a rel. efficiency for Ag K $\alpha$  of about 45% ). This good efficiency, together with the energy resolution of 250 – 300 eV for Mn K $\alpha$  X-rays ( 4 times better than a gas proportional counter ) make today's PIN- diodes a practical detector for coating thickness and material analysers.

### Examples - comparison of spectra

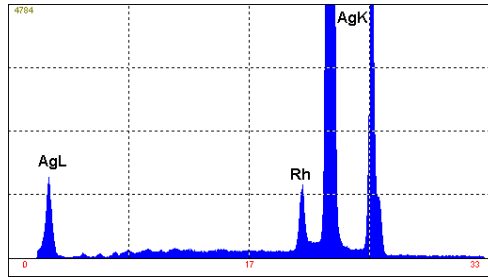
The examples show a comparison of spectra generated with both types of detectors. The samples used are identical. Two ComPact 5 instruments were used. One equipped with a Xe- filled proportional counter, and the other with a  $25\text{mm}^2$ , 0,5 mm thick PIN- diode. The measurement time was 30 seconds, and the HV = 40 kV in both cases. The difference was that the ComPact 5 equipped with the proportional counter had a 0,2 mm $\varnothing$  beam size while the ComPact 5 with the PIN diode had 0,5 mm $\varnothing$  beam size. It could be clearly seen that due to the better spectral resolution the peak/ background ratio for the PIN- diode is much better, leading to a lower minimum limit of detection. The spectrum of the lead- free solder, containing 0,7% of Copper, clearly shows the better MLD. In cases where the composition of the sample is known, it is possible to achieve very good and accurate results with proportional counters by applying mathematical enhancement techniques to the spectra. In the ComPact 5 for instance a mathematical peak deconvolution technique is used giving good and stable results even at overlapped peaks. But if the composition of the sample is unknown and the number of elements is  $>8$  it will be very difficult to find some overlapped or hidden elements, in particular if their concentration is low. This is clearly the great advantage of the PIN- diode detector.

# Application Note

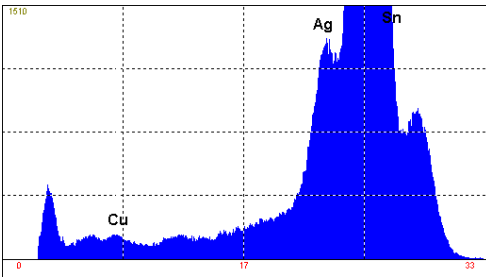
Below are some examples that demonstrate the advantage of the PIN-diode detector. Even though the mathematical spectrum processing will produce a correct result with the proportional counter it will be very hard to find some of the elements if the composition is unknown.



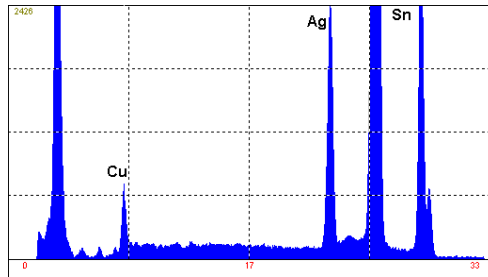
0,29µm Rhodium on Silver  
System with Proportional Counter



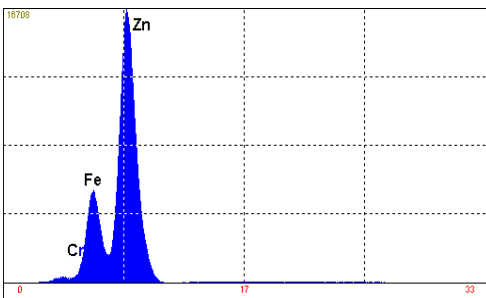
0,29µm Rhodium on Silver  
System with PIN-Diode



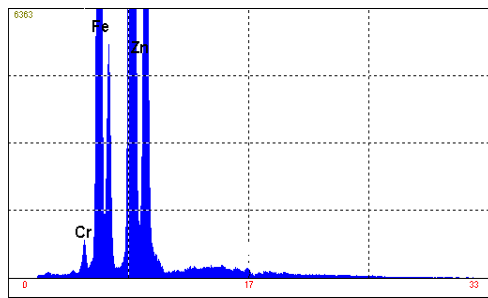
Lead free Solder, SnAgCu with 0.7% Copper  
System with Proportional Counter



Lead free Solder, SnAgCu with 0.7% Copper  
System with PIN-Diode



Screw, yellow chromate overcoat  
System with Proportional Counter



Screw, yellow chromate overcoat  
System with PIN-Diode

## Conclusion.

Modern Peltier-cooled silicon PIN-diode detectors have reached a level of performance by now that makes them practical detectors for a variety of analytical tasks in material and coating thickness X-ray analysers. Only in cases where the elements are known, a very small spot size ( $< 200\mu$ ) is needed and an analysis time of less than 30 sec. is required a gas proportional counter is still the best choice.

  
Application

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