

Silicon Drift Detector Spectrometers for High Count Rate XRF and TXRF Applications

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A 50 mm² silicon drift detector (SDD) has been successfully applied to TXRF (total reflection x-ray fluorescence) and other XRF applications. The SDD offers a large solid angle, excellent energy resolution (<130 eV FWHM at 5.9 keV, 4-6 μ s peaking time), and high count rate performance (> 600 kcps throughput at 0.25 μ s peaking time), all of which are essential performance requirements for many XRF applications. The Vortex[®] spectrometer has recently been applied in synchrotron radiation TXRF (SR-TXRF) astrophysics applications, at the Stanford Synchrotron Radiation Laboratory (SSRL) in the analysis of the chemical composition of solar wind particles. This paper describes these measurements and the advantages of the Vortex[®] performance in achieving these results.

1.0 Introduction

A 50 mm² silicon drift detector (SDD) has been successfully applied to TXRF (total reflection x-ray fluorescence) and other XRF applications. The SDD offers a large solid angle, excellent energy resolution (<130 eV FWHM at 5.9 keV, 4-6 μ s peaking time), and high count rate performance (> 600 kcps throughput at 0.25 μ s peaking time), all of which are essential performance requirements for many XRF applications [1-5]. The Vortex[®] 50 mm² SDD family of products is shown in Figure 1. The Vortex[®] spectrometer has recently been applied in synchrotron radiation TXRF (SR-TXRF) astrophysics applications, at the Stanford Synchrotron Radiation Laboratory (SSRL) in the analysis of the chemical composition of solar wind particles. This paper describes these measurements and the advantages of the Vortex[®] performance in achieving these results.



Figure 1. Examples of the Vortex[®] 50 mm² SDD family of products, shown with the power supply and digital pulse processor.

2.0 Vortex[®] SDD Performance Summary

Traditionally, low noise x-ray spectroscopy, and specifically TXRF, has utilized a low noise Si(Li) detector. Si(Li) detectors are typically large and cumbersome, due to the requirements of the cryogenic design and the large liquid nitrogen (LN₂) dewar for cooling the detector. In addition, only a small active area Si(Li) detector (~10 mm²) can achieve the required energy resolution for many x-ray spectroscopy applications and in order to achieve the optimum energy resolution (~130 eV FWHM) very long amplifier peaking times must be used (32 - 64 μs). This situation is primarily due to the fact that the Si(Li) detector consists of a geometrical structure which results in an input capacitance directly proportional to its active area size, which is typically on the order of 1 pF for 10 mm² area detectors. This relatively large capacitance requires that the system be operated in the long amplifier peaking time regime in order to achieve the best energy resolution. In contrast, the input capacitance of an SDD structure does not depend on the detector active area, and the large active area 50 mm² SDD has an input capacitance of only ~0.06 pF. This very small capacitance means that an extremely good energy resolution (for example 125 eV FWHM) can be achieved at 6 μs peaking time (at 5.9 keV), as shown in Figure 2. In addition, this excellent energy resolution can be achieved at much higher temperatures than required for Si(Li) detectors, thus the need for LN₂ is eliminated, and the spectrometer can be designed in a very compact, flexible geometry to suit many different applications. Figure 3 shows the high count rate performance of the 50 mm² Vortex[®] SDD, as a function of amplifier peaking time.

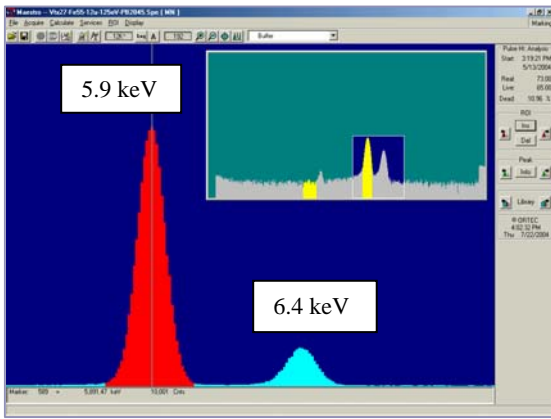


Figure 2. Typical spectral response of the Vortex[®] to ⁵⁵Fe: energy resolution is 125 eV FWHM at 5.9 keV and 6 μs peaking time.

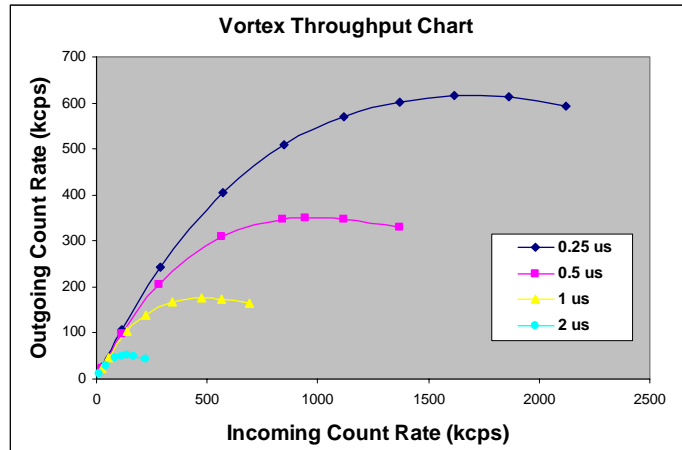


Figure 3. Outgoing count rate as a function of incoming count rate and amplifier peaking time, for the Vortex[®] system.

For TXRF applications, there are clear advantages of the Vortex[®] SDD compared with conventional Si(Li) detectors, starting simply from the detector area and spectrometer geometry. The compact size of the Vortex[®] SDD allows for a very simple mechanical arrangement of the spectrometer that yields the high angular precision required for TXRF measurements. The elimination of the LN₂ dewar greatly simplifies the operation within the clean TXRF environment. TXRF requires the best performance in energy resolution in order to achieve the required detection limit, which is set by the total number of counts measured. The Vortex[®] offers a much higher count rate capability compared with a Si(Li) detector, without compromising energy resolution. The 2-3 times higher count rate capability, with excellent energy resolution, leads to an improved detection limit by approximately the square root of two.

3.0 TXRF on the Genesis Mission Samples

The Vortex[®] spectrometer has been applied to SR-TXRF measurements of solar wind particles collected on the NASA Genesis Mission. One of the primary goals of this mission was to obtain accurate measurements of solar elemental abundances in order to ultimately gain a better understanding of the origins of the planets. Extremely clean germanium, silicon and sapphire wafers were installed on the Genesis satellite and were exposed to solar wind for 27 months. Solar wind is predicted to have 1 keV of kinetic energy per atomic mass unit, so 75 keV arsenic ions in silicon will implant to a depth of ~55 nm with a FWHM distribution of 35 nm. For 31 keV phosphorus ions in silicon, the implantation depth is ~42 nm with a FWHM distribution of 46 nm. The expected solar wind fluences are $\sim 1 \times 10^{11}$ atoms/cm² for Ni and 3×10^{12} atoms/cm² for Fe, with most other transition metals having significantly smaller fluences.

Unfortunately, the Genesis space craft crashed to earth upon its return, due to a malfunction of the parachute. The crash caused the original 100 mm diameter wafers used to collect the solar particles to break into many small pieces. The broken wafers were also contaminated with dirt from the Utah desert crash site. This has, of course, added significant complications to these studies. For example, all the small pieces had to be sorted and separated as to substrate type, and samples large enough for measurement had to be cleaned. Since TXRF is also very well suited to studying sample cleanliness, we have been able to contribute to this aspect of the work as well. Since most of the samples were broken into pieces smaller than 1 cm², the only samples suitable for the TXRF analysis were the sapphire samples, which provided the largest pieces for analysis and had the cleanest surfaces. In fact, pieces of the more fragile Ge samples could not even be found. The only evidence of the Ge wafers was a monolayer of Ge contaminant on the surfaces of all the other samples.

Figures 4 and 5 show spectra collected with the Vortex[®] SDD from one of the sapphire samples, at two different angles of incidence. The lower angle of incidence (the 0.12 degree spectrum shown in Figure 4) shows significant levels of surface contamination, including the Ge dust. At the higher angle of incidence (0.5 degree spectrum shown in Figure 5) most of the surface contamination is suppressed by increased background and the spectrum is dominated by bulk contaminants. These spectra showed the high degree of contamination present on these samples and served as a starting point for the subsequent sample cleaning that had to be done before meaningful data could be extracted.

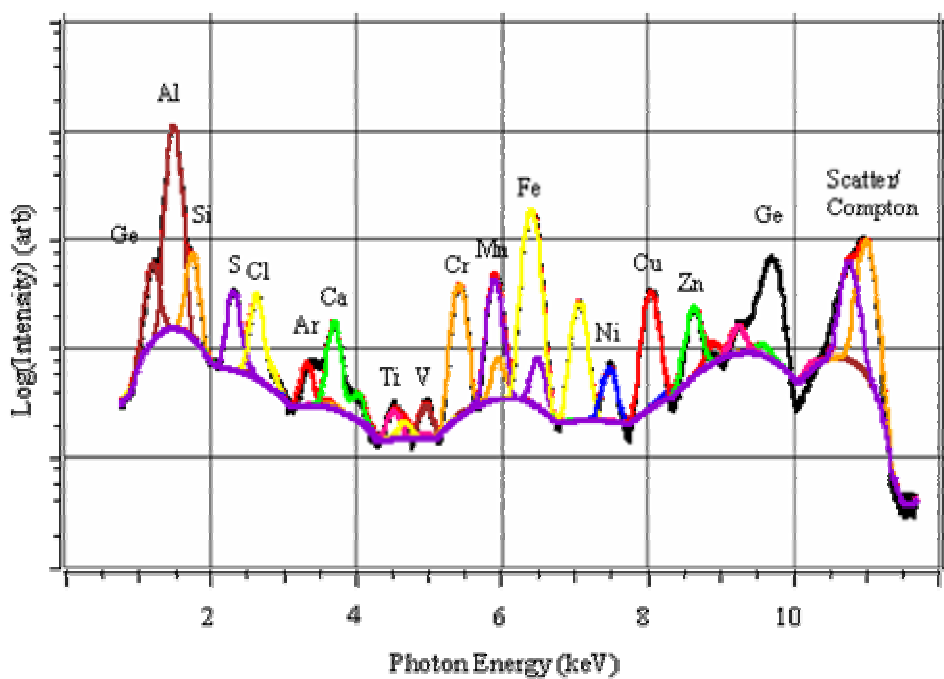


Figure 4. Genesis flight sapphire sample #E50651; data collection angle of incidence is 0.12 degree; 11 keV SR beam. Surface contamination elements, including Ge are observed.

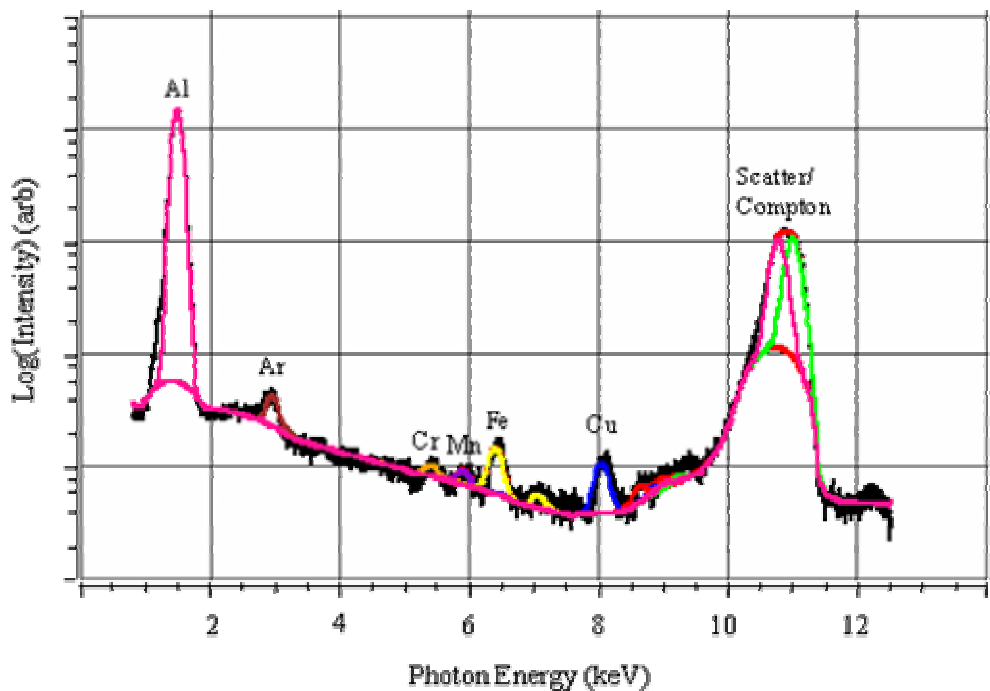


Figure 5. Genesis flight sapphire sample #E50651 (same sample as in Figure 4); data collection angle of incidence is 0.5 degree; SR beam 11 keV. The higher angle of incidence significantly reduced the contribution from the surface contamination, thus allowing the species present in the bulk to be measured.

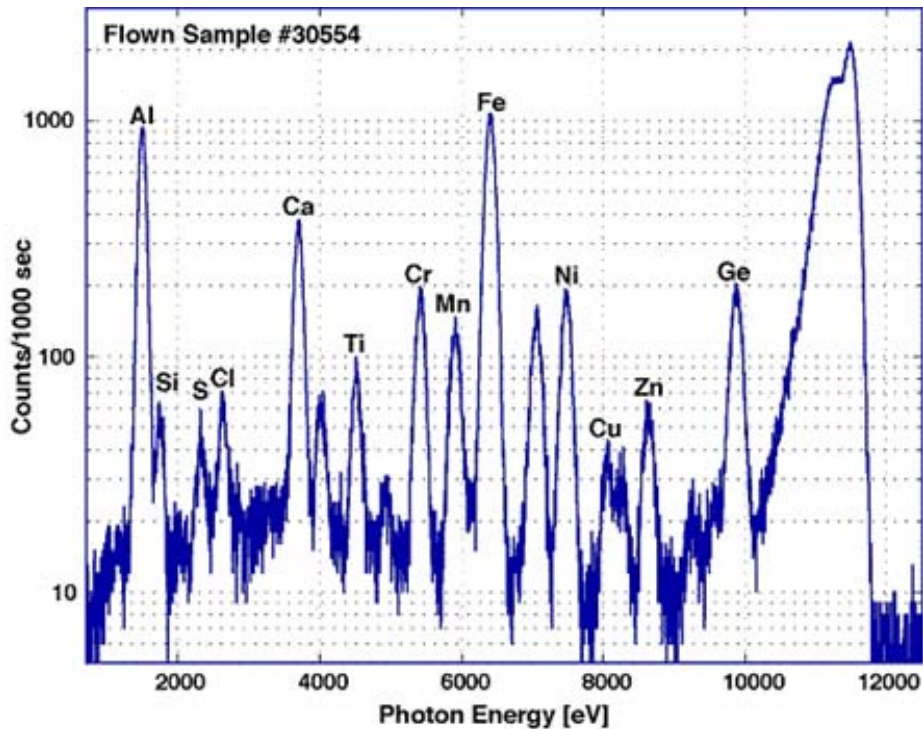


Figure 6. Genesis flight sapphire sample #30554; x-ray spectra after sample cleaning, showing evidence of solar wind particles embedded in the sapphire substrate.

The TXRF spectrum from another Genesis sapphire sample is shown in Figure 6. The sample was cleaned using a variety of physical and chemical methods, including megasonic cleaning, UV-ozone, and chemical etching, which resulted in the removal of a significant fraction of the contaminants. Because of the large number of peaks in the Genesis samples, we used the scatter peak to normalize the spectra to the standard. Using this method, we obtained the following quantities for this sample (all in units of atoms/cm²): Al- 9.9×10^{15} , Si- 5.0×10^{13} , S- 2.75×10^{12} , Cl- 1.85×10^{12} , Ca- 2.41×10^{12} , Ti- 2.27×10^{11} , V- 1.2×10^{10} , Cr- 2.95×10^{11} , Mn- 1.43×10^{11} , Fe- 1.13×10^{12} , Ni- 1.21×10^{11} , Cu 1.50×10^{10} , Zn- 2.51×10^{10} , Ga- 4.7×10^9 , and Ge- 7.5×10^{10} atoms/cm². For this unique experiment, the Vortex[®] SDD, with its good peak-to-background ratio, resulted in an excellent detection limit of 1×10^8 atoms/cm², which defines the state-of-the-art for this technique.

These measurements could not have been accomplished with a conventional Si(Li) detector, due to the compact size of the clean mini-environment of the test setup. The necessity to fill an LN₂ dewar would have compromised the cleanliness of the experimental setup. In addition, the high count rate and commensurate excellent energy resolution of the Vortex[®] SDD resulted in an unprecedented detection sensitivity, as detailed previously.

4.0 Conclusions

4.1 The Vortex[®] SDD is a superior spectrometer and can replace the conventional Si(Li) detector for SR-TXRF and commercial TXRF applications. With its excellent energy resolution, high count rate capability, large active area, and maintenance-free operation, the Vortex[®] SDD offers clear performance advantages over the Si(Li) detector for TXRF applications.

4.2 For the performance conditions required by the SR-TXRF measurements, the Vortex[®] SDD is 2-3 times faster than the Si(Li) detector, and resulted in unprecedented detection limits of 1×10^8 atoms/cm², which defines the state-of-the-art for this technique.

4.3 The compact size and elimination of LN₂ is a very important aspect for these types of TXRF studies which are performed in a mini-clean room environment. The smaller design allows for a much better control of cleanliness, and also facilitates the precision motions required to align the samples and test chamber.

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