

SEM/EDS Analysis of Cell Phone Cover Glass Facilitated by the Use of a Silicon Drift Detector

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The Silicon Drift Detector (SDD) was first conceived of about 30 years ago and has largely supplanted the Si(Li) detector[1]. The current generation of commercially available SDD meets or exceeds the performance of a Si(Li) detector in nearly every way. The purpose of this work is to illustrate how an SDD applied to the analysis of a set of glass samples provides more detailed information in roughly the time it took to collect a single spectrum with a Si(Li) detector.

The samples are pieces of cover glass from popular cell phones. Glass samples from different years were inspected. This is a challenging sample as it contains Na and Na in glass is mobile. If the electron beam is left in one spot at too high an intensity the measured Na intensity will quickly drop off as Na ions migrate away from that location.

Some of these samples are characterized by a locally high concentration of K near the surface[2]. The analysis will reveal this distribution.

Pieces of broken glass were removed from three cell phones. The samples were mounted in epoxy and polished to a smooth surface so that they could be viewed in cross section. The samples were carbon coated after polishing.

With the Si(Li) detector a spectrum was typically acquired at about 2,000 cps storage rate and 30% dead time for about 100 - 200s live time or clock time[3][4]. The SDD counts x-rays more quickly under the same conditions as does a Si(Li) detector and can count even more quickly if the beam current can be increased[5]. Moreover, SDDs are available providing much more solid angle while maintaining approximately the same resolution. Larger solid angle allows one to reduce the beam current which reduces the likelihood of Na migration or charging in these samples while still collecting x-rays at a high rate.

Table 1 shows the sensitivity of two different SDDs in counts per second per nanoAmp. One has 30mm² active area and one has 100mm² active area but with a rectangular design allowing it to be positioned much closer to the sample which greatly increases solid angle. In addition, this is a windowless detector.

It is suggested here that the analyst take advantage of the increased throughput of the SDD to gain new insights into the sample and to improve the confidence one has in the result by collecting multiple spectra in a comparable time as when using a Si(Li) detector to collect one spectrum.

Figure 1 shows the plot of the counts from 11 spectra taken at the same location for 10s live time each while the beam was rastered in a small circle about 10 microns across. This revealed the Na intensity dropping off after about 50 seconds at the lower current but almost immediately at the higher current. Repeating this at a few different beam currents allows the analyst to quickly find the safe operating conditions for analyzing any glass sample. 110s total live time was required for each test.

Figure 2 charts the composition of K across a sample taken at nine locations. It reveals the K distribution. Collecting multiple spectra reveals compositional variation in a sample when it may not be visible in the SE or BSE image. Using selected areas rather than points for each analysis reduced the possibility of Na dispersion and can allow for the user of a higher beam current.

Acquiring a single spectrum provides no context. Acquiring many spectra at one location provides a time series that reveals the loss of material due to ion mobility or sample decay or the accumulation of contaminants. Acquiring a number of spectra in a region provides a statistical view of the uniformity of the composition of the sample in that region. Acquiring a number of spectra from points arrayed in a line provides a line profile. All of these can be performed today in about the time it used to require to collect a single spectrum.

References:

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- [5] D. E. Newbury, N. W. M. Ritchie, Materials Science and Technology, (2016), p. 1017.

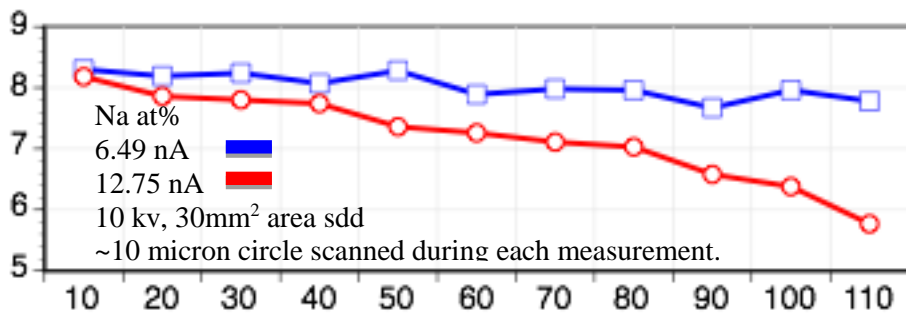


FIGURE 1. time series measuring na. not that at the lower beam current the na is stable for about 50s. Scale is in seconds.

Area	counts / sec / nA
30 mm ²	2,867
100 mm ²	37,643

Table 1. Si K line counts measured on glass with two SDDs. The larger detector gains about 3x sensitivity in size and about 4x sensitivity because the shape allows it to be much closer to the sample. Also, it is a window-less detector.

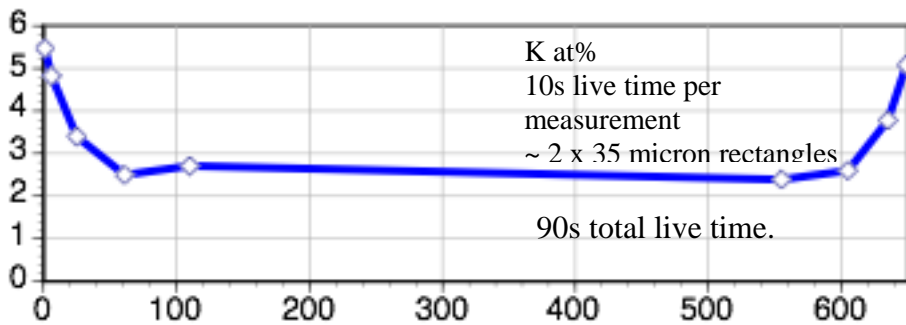


Figure 2. Line profile calculated from 9 measurements of rectangular areas across a 650 micron wide glass sample. Scale is in microns.