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SEM EDS Mapping of Ultra-Low Energy X-rays Using a Silicon Nitride Window Silicon Drift Detector

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The ability to detect ultra-low energy X-rays (<100 eV) is considered a benchmark for the sensitivity of Silicon Drift Detectors (SDD). It has long been recognized that detecting Si L line at 92 eV requires the most sensitive windowless Energy Dispersive Spectroscopy (EDS) detectors. However, silicon nitride window EDS detectors with fast and low noise pulse processors can routinely detect Si L. The same is true for detecting Al L, which is even lower at 72 eV. By adding a sealed ultra-thin silicon nitride window in front of the detector module, it increases low energy X-ray transmissivity relative to a traditional polymer window, as well as keeps the detector cooled and under vacuum at all times. This windowed design eliminates the risk of detector contamination during venting cycles, supports plasma cleaning, and allows for the use of variable pressure mode.

Al and Si are adjacent elements in the periodic table and abundant in various types of materials, such as geological materials, alloys, and semiconductors. The Al L peak at 72 eV is much weaker in intensity than the Si peak and is more difficult to detect due to the absorption of X-rays in the window. Figure 1 shows Al K and Al L maps collected at 25, 5, and 1.5 kV accelerating voltages from an unpolished semiconductor sample using an EDS detector with silicon nitride window. The Al L line can be detected even with accelerating voltage of 25 kV. Due to the surface sensitivity of the low energy X-rays, the map shows the surface structure of an aluminum pad (Figure 1b), whereas the higher energy Al K line with 6 µm emission depth [1] resolves the buried structures (Figure 1a). The aluminum pad in the L line map is smaller than that in the K line map because the nitride barrier layer around the pad blocks all the Al L X-rays beneath it and signals only escape from the pure aluminum surface. Therefore, at relatively high accelerating voltages, mapping with the Al L line is practical and useful to represent different depth information of the sample than with the Al K line. At 5 kV accelerating voltage, the emission depth of the Al K signals reduces to 0.34 µm [1] and is less than the thickness of the nitride layer. Figure 1c and 1d indicate that the Al K and Al L signals come from approximately the same region. Extremely-low kV EDS mapping can effectively enhance surface sensitivity and reduce beam damage to the sample, but it limits the energy lines available in the EDS spectrum. In this example, there is no sufficient overvoltage to excite the Al K line at 1.5 kV accelerating voltage and using the L line is the only option to map the pad structure (Figure 1e).

Because it is common to have Al and Si phases coexist in a sample, separating Al L and Si L lines is desired. The theoretical resolution for Si and AL L lines is on the order of 40-50 eV, while the peak separation is only 19 eV. This means that the two peaks are always overlapped in an EDS spectrum. Since there is very little theory and data available regarding excitation and emission efficiency, deconvoluting the peaks is not practical with current stage of modeling and detector technology. In principle, using a Region of Interest (ROI) on the lower energy side of the Al L peak will separate the two peaks. However, the Si L peak is much more intense than the Al L peak, so the tail of the Si peak will be dominating even at the 40-50 eV energy range. But with a silicon carbide sample, the Si L signal is attenuated by the carbon, and it becomes possible to separate the two regions using the ROI. Figure 2 shows EDS maps of a piece of aluminum tape on a silicon carbide chunk collected at 2 kV accelerating voltage using a silicon nitrite window EDS detector. Al L and Si L regions are separated out using this ROI method.

In conclusion, mapping Si L and Al L lines is a routine practice for silicon nitride window EDS detectors with fast and low noise pulse processors. Coupled with the K line maps, the L line maps provide different depth information of the sample at relatively high accelerating voltages. At extremely low voltages, map data can still be collected using the L lines, whereas there is no longer access to the K lines. Even separation of these two ultra-low energy peaks with 19 eV energy difference in the map is possible.

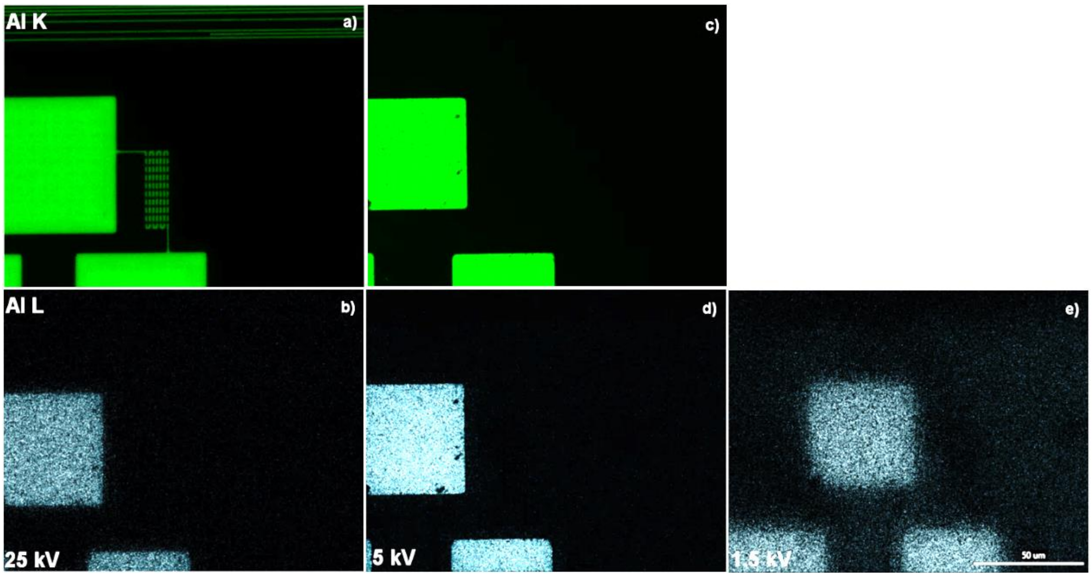

Figure 1. Al K (top) and Al L (bottom) maps of an unpolished semiconductor sample collected with a silicon nitride window EDS detector at 25 kV (left), 5 kV (middle) and 1.5 kV (right).

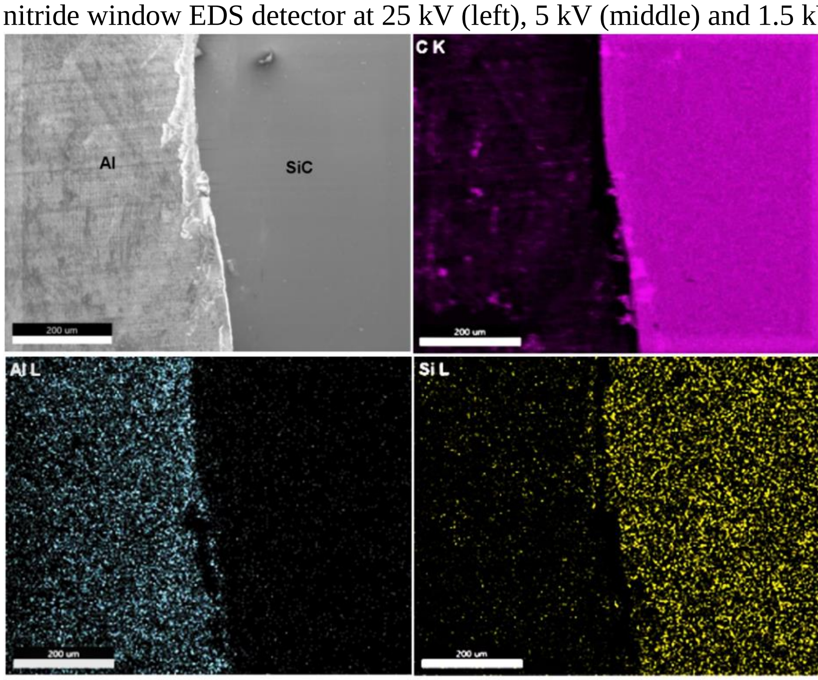

Figure 2. Secondary electron image and C K, Al L, and Si L maps collected from a piece of aluminum tape on a silicon carbide chunk at 2 kV using a silicon nitride window EDS detector.

Reference:
[1] NWM Ritchie, NIST DTSA-II software, https://cstl.nist.gov/div837/837.02/epq/dtsa2/ (accessed February 22, 2022).

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