

Detection of Lithium X-rays by EDS

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Recent advances in hardware technology for EDS have improved detection capability in many areas. Large area detectors with good resolution, made possible by SDD technology, have increased sensitivity so that acceptable count rates can be obtained at the low accelerating voltages. One of the less heralded areas is the progress in the efficiency of the detection of very low energy X-rays. While the detection of Be K α using Si(Li) technology in SEM was first reported almost 30 years ago, it was only a few years ago when SDD had progressed sufficiently to detect BeK α . However, progress has continued and we are now able to report latest progress in this area. This study focuses on detection and measurement of very low energy X-rays (150eV and below), in particular the detection of LiK α X-rays at 54eV.

Low noise sensor electronics and enhanced charge collection efficiency have improved sensitivity to SiL ℓ at 92eV. The improvements achieved have resulted in clear SiL ℓ and AlL ℓ lines being commonly observed in materials such as Al-Si alloys using 80mm² and 150mm² area detectors. The other significant barrier to detection is the poor transmission of very low energy X-rays through the detector vacuum window. Even the latest polymer technologies offer little usable transmission below AlL ℓ . To test the potential of the latest sensor package designs, a number of special detectors have been made using a windowless design, to maximize low energy sensitivity.

To test the feasibility of this detector technology for the detection of X-rays from lithium, a sample of Li metal was prepared. The sample was cut from a Li metal rod in a bag pressurised with pure N₂ to minimise surface contamination through reaction with air, before rapid transfer to the load lock of the SEM. A spectrum collected from the sample shown in Fig. A, shows a pronounced peak at around 54eV, well above the X-ray background and clearly separated from the triggered noise peak. Other peaks, particularly OK α , but also CK α , NaK α and in some areas NK α were also detected. The OK α is always higher than the Li peak, but this is to be expected because the efficiency of the detector at 500eV is many times that at 50eV, even with this special detector configuration.

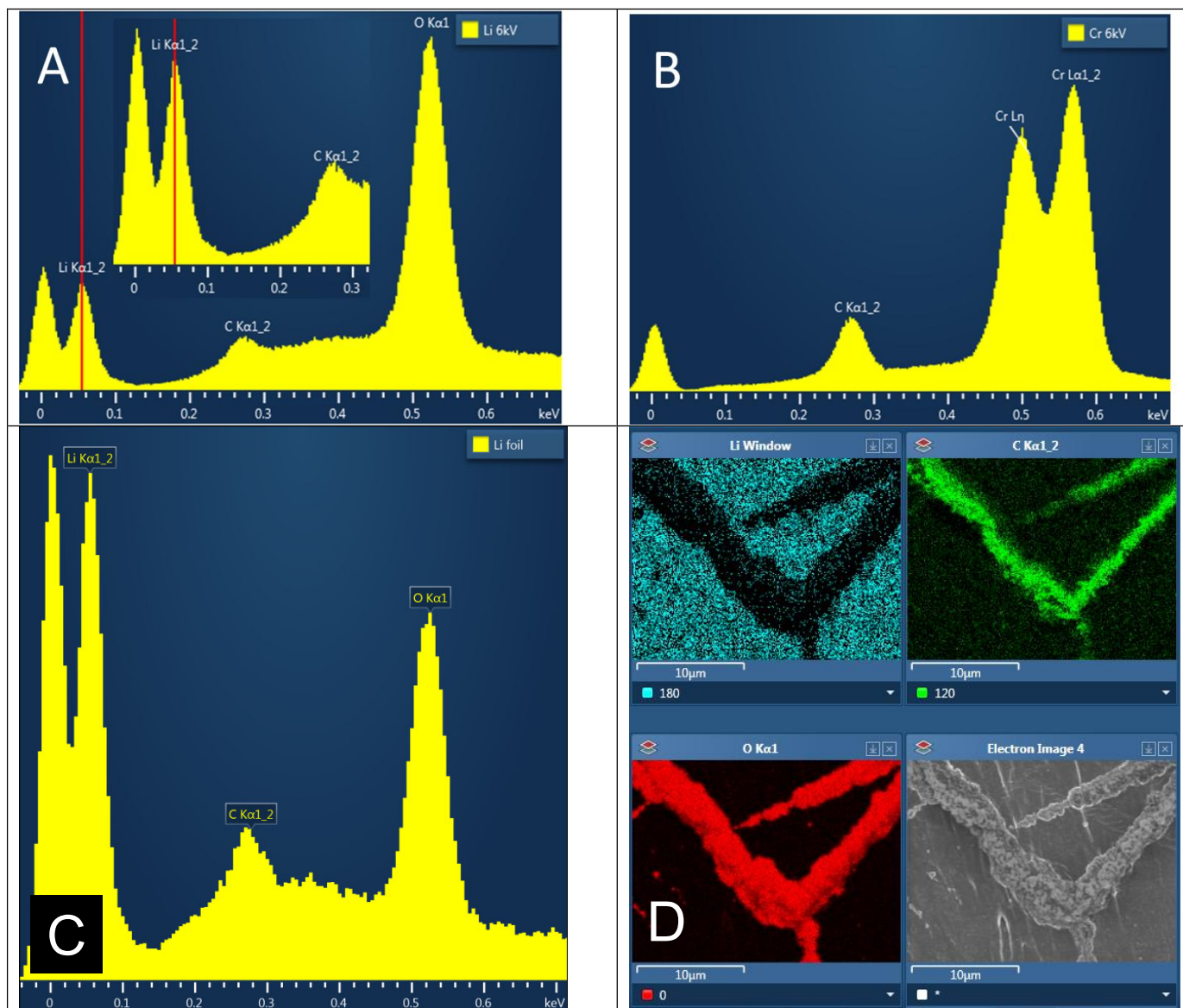
To make sure that the observed Li peak was not some artifact of the excitation or detection system, spectra were collected from a number of other materials with no Li content using exactly the same microscope, detector and electronic configuration. No spurious signal was seen in the range 40 – 70 eV in any spectrum other than that from the Li metal, providing strong evidence that the peak near 54eV detected in the spectrum from Li metal is indeed due to the LiK α characteristic X-ray emission. Fig. B shows the spectrum obtained from Cr metal where there is clearly no low energy peak at the LiK α energy.

Li metal is reactive to a number of gases that the sample was exposed to during preparation including N₂ to form Li₃N and atmospheric water vapour to form LiOH [1]. Therefore further tests have been conducted on a Li foil sample that was prepared, and loaded through a special microscope loading

process under vacuum and argon at Hitachi High Technology Corp., Katsuta, Japan. Spectra collected from this sample (Fig. C collected at 2.5kV) show significantly lower levels of $OK\alpha$, allowing lower kV to be used. The benefit of this method was shown by exposing this sample to air for 10 minutes. This exposure results in a rapid alteration to oxygen and carbon containing species, preferentially along grain boundaries, as shown by X-ray maps collected for $LiK\alpha$, $CK\alpha$ and $OK\alpha$ after exposure (Fig. D).

Reference

[1] U. Wietelmann, R. J. Bauer (2000), Lithium and Lithium Compounds. Ullmann's Encyclopedia of Industrial Chemistry, Wiley.



Figs. A. Spectrum collected from corroded lithium metal at 6kV. B. Spectrum collected from chromium metal under identical conditions to the spectrum in Fig. A. C. Spectrum collected from a clean Li foil at 2.5kV. D. X-rays maps for $LiK\alpha$, $CK\alpha$, and $OK\alpha$ showing grain boundary alteration in the foil after exposure to air.