Low Energy XEDS Analysis and Light Element Detection

Greaves, M.[1], Lathabai, S.[1], Veitch, C.[1] and de Vries, M.[1]

[1] CSIRO Manufacturing, Australia

New production methods such as additive manufacturing (more commonly known as 3D printing) have yielded some interesting challenges for material characterisation and analysis. Low concentrations of nano-sized constituents, combined with different and varying microstructures compared to traditionally cast material and the variations resulting from different printing parameters; make characterisation of these products very challenging. Recently, CSIRO Manufacturing purchased an Oxford Instruments "Extreme" low energy X-ray detector (the first of its type in Australia) and this has opened up a new window on elemental microanalysis. A re-designed collimator and electron trap, allow for short working distance to be employed and a larger detector (100 mm^2) and improved electronics improve the signal and processing speed. The windowless detector is also designed to work at lower incident electron beam energies (< 7 kV). These attributes combined, enable users to acquire data that is significantly more surface sensitive, spatially resolved or detect elements down to and including lithium. This makes it ideal for characterising and analysing new materials to give insights to the manufacturing process and product performance. The detector is installed on a Zeiss Merlin FESEM (Field Emission Scanning Electron Microscope). An Oxford Instruments X-Max (80 mm^2) conventional X-ray SSD detector is also installed on it allowing direct comparisons between the two detectors possible.

An aluminium alloy (AlSi10Mg) was additively manufactured in CSIRO's Lab 22 (Australia's Centre for Additive Innovation assisting the manufacturing industry to prosper through innovation). The printed alloy in this work was formed using Selective Laser Melting. Layers of powder are built upwards sequentially with successive laser melting. Changing process parameters lead to variations in microstructure, resulting in varied product properties. The surface of the alloy was analysed using both the Extreme and X-Max detectors. An incident beam energy of 3 kV was used when analysing with the Extreme detector and energies of 5 kV, 10 kV and 20 kV used when analysing with the X-Max detector. The Extreme detector showed the presence of both copper and iron impurities localized in discrete structural areas, which can impact on the performance of the alloy. Characterisation is essential to aid process optimisation in terms of porosity distribution, density and ductility of the product enabled by investigating morphology and grain refinement depending on application requirements.

Spectra collected by the X-Max detector at 20 kV showed no evidence of the k-lines of either copper or iron. There were small peaks in the spectra generated from this detector at 5 kV indicating presence only at the surface when considering increased electron interaction volume penetration effects with increasing beam energy. Resultant X-ray maps from this sample show this effect very clearly and highlight the importance of selecting the correct X-ray line and beam energy to use in analysis. With the Extreme detector, 20 nm impurity particles were identified along grain boundaries and iron detected within the ternary phase of the aluminium matrix which were both indiscernible with the traditional detector.