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Energy Dispersive X-ray Spectroscopy in the Transmission Electron Microscope – Precision and Accuracy.

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The relative ease of using the EDS technique to analyse the elemental composition of specimens has meant that this characterisation technique is almost ubiquitous with electron microscopy. However, the depth of analysis widely ranges from qualitative to "semi-quantitative" through to quantitative. The approach to data collection and analysis will depend on the researchers' final goal; are they looking to detect the presence or otherwise of a particular element (qualitative EDS), do they need to confirm a particular mineral phase has been formed (semi-quantitative EDS), is there an exact stoichiometry that has been targeted (quantitative EDS).

It is possible to achieve a precision of measurement better than 1 % without too much work. This precision is dependent on the x-ray counts collected with high counts being readily attainable for many elements even when nano-metre thin TEM specimens are used. How "quantitative" this data is depends on the x-ray counts, the number of grains sampled, along with the relativeness of the standards to the measurement of the unknown.

Another limiting factor for EDS quantitative analysis in the TEM is x-ray absorption. This is a most significant influence to x-rays of ~ 1 keV or less making analysis of elements like oxygen difficult. It is however still possible to achieve quantification of the lighter elements with a little extra work.

With a focus on using k-factors for calibration the advantages / disadvantages of this technique will be discussed along with a few easy to follow tips on how best to quantify EDS data in the TEM.

The precision of EDS measurements will be discussed with examples of how thickness, density and crystal structure may all create varying levels of deviations within the data. Further to this, examples of how crystal structure related channelling can affect the x-ray signals, and a novel approach, amorphisation can be used to rectify this potential issue.

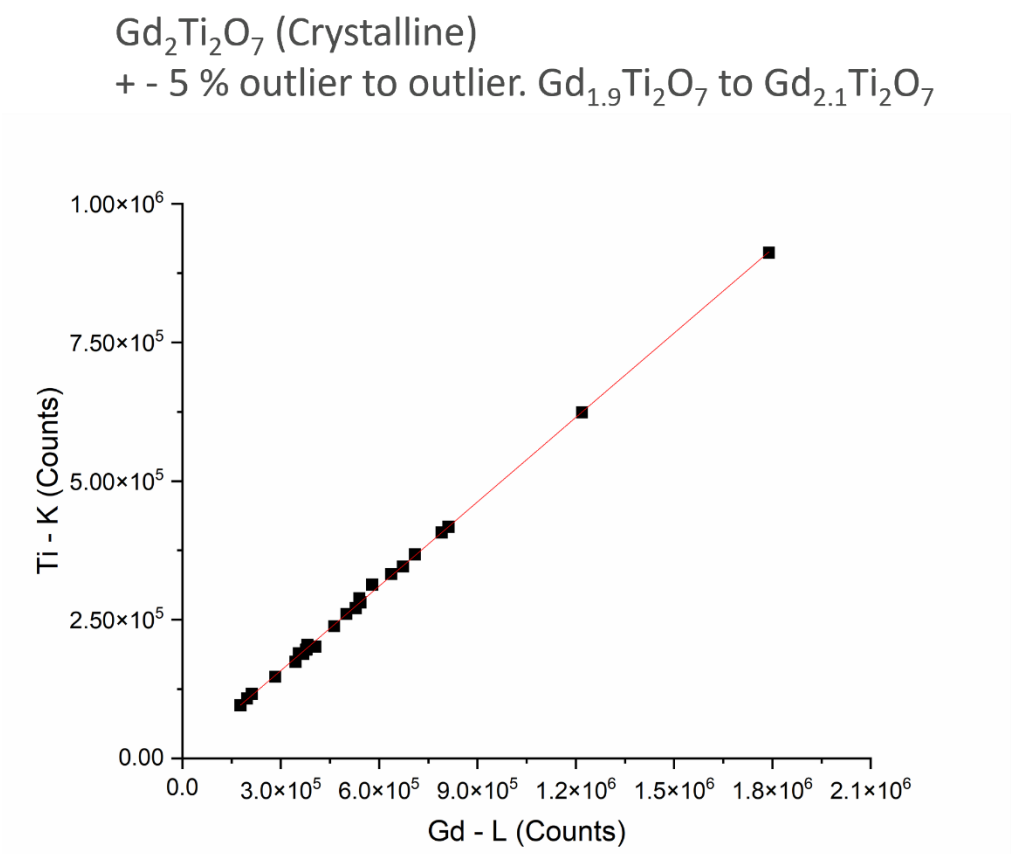

Fig. 1. *A plot of EDS x-ray intensities, taken from 25 grains of pyrochlore $Gd_2Ti_2O_7$, for gadolinium-L peak versus titanium-K peak. A linear fit is shown to highlight the precision between measurements.*

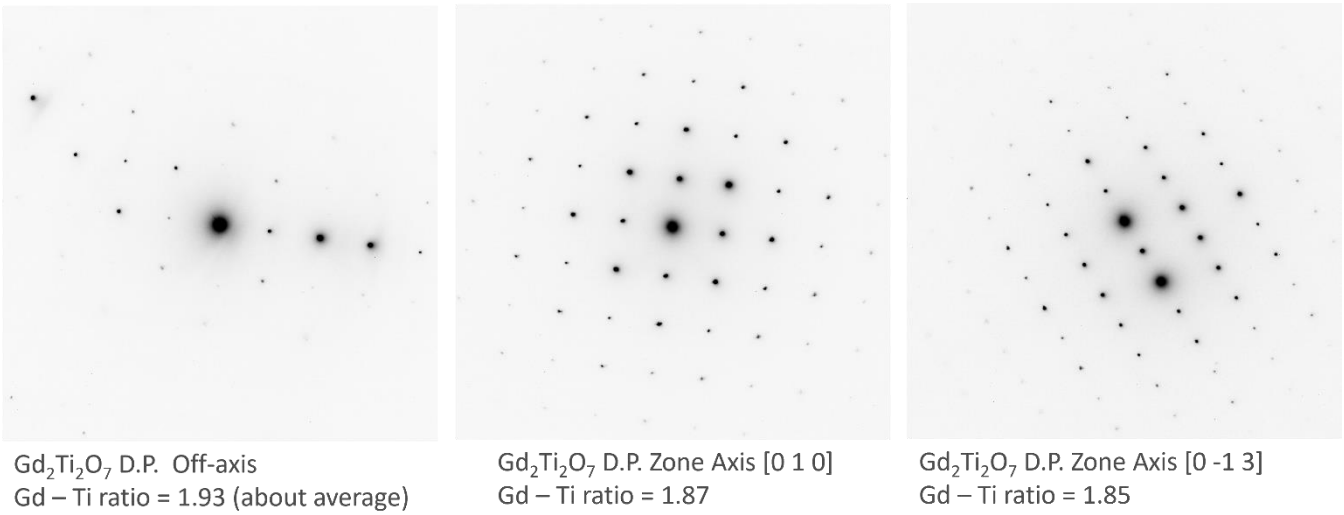

Fig. 2. *A selection of electron diffraction patterns from the same grain, pyrochlore $Gd_2Ti_2O_7$, taken at 3 different angles. The zone axis and ratios of Gd:Ti x-ray peak intensities are given below each DP.*