doi:10.1017/S1431927620013264
Microsc. Microanal. 26 (Suppl 2), 2020


# FEGSEM X-Ray Mapping System with Multiple SDDs for Quantitative X-Ray Mapping and Imaging

Richard Wuhrer[1] and Ken Moran[2]

[1]Western Sydney University, Penrith, New South Wales, Australia, [2]Moran Scientific Pty Ltd, Bungonia, New South Wales, Australia

A JEOL Field Emission Gun Scanning Electron Microscope (FEGSEM) system that has been retrofitted with three Amptek Silicon Drift Detectors (SDD's) positioned around the chamber for combined detector X-Ray Mapping (XRM), Figure 1. The reasons for this retrofit are that the FEGSEM has 1) a totally clean vacuum system utilising scroll vacuum pumps and a turbo-molecular pump; thus minimizing contamination, especially on the windowless detector, 2) the ability to operate the FEGSEM at lower accelerating voltages with lower beam currents and small spot size, 3) a larger chamber for additional modifications, 4) excellent beam stability for extensive XRM operating 24 hours-7 days per week [1, 2] and 5) a STEM detector for planned STEM XRM.

The dedicated XRM system has three Amptek SDD's, with 25mm$^2$ detector area and 9 inch vacuum extensions. The detectors are an Amptek FAST123 SDD's with one detector having a $Si_3N_4$ ultrathin C1 window (150nm), another with $Si_3N_4$ super ultrathin C2 window (40nm) and the third is a windowless detector. The longer detector vacuum extension allows for closer proximity to the sample allowing greater collection efficiency. The operating software is the Moran Scientific X-Ray Mapping (XRM) package.

Quantitative X-ray mapping (QXRM) using multiple detectors, reduces mapping time and also improves the ability to map minor and trace elements accurately. One interesting outcome is the ability to map at much lower beam currents [2-5], thus allowing less sample damage. This has become easier due to the release of the very large area detectors (70mm$^2$) and now 160mm$^2$.

Our research aims at developing post processing techniques to improve the quantitation of X-ray map data and to develop further post processing techniques for improved characterisation as an aid in assessing the practical properties of complex materials [6-8]. This also includes developing techniques of handling X-ray mapping data collected from multiple X-ray detectors spaced around the column. One technique developed, called "Colouring Verification Technique (CVT)", and involves assigning a different RGB colour to each detector for the same element. When combining the three maps of the same element, a grey scale map should be obtained, indicating a total correlation between the three detectors at the most critical final stage of quantification [2-3].

Figure 2a shows the combined X-ray maps for Iron from the three different detectors located around the chamber and Figure 2b shows the combined X-ray maps for Oxygen from the three different detectors. As can be seen, the x-ray maps from the higher atomic elements show a flatness and only two colour variation depending on orientation. The Oxygen x-ray maps, being lighter elements show many colour variation and also reveal light element quantification can be a major concern with quantifying rough samples.

This presentation will cover the current modifications carried out and results being obtained as well as some future modifications in progress, such as 1) Peltier cooled device in chamber for minimising further contamination, 2) plasma cleaner, 3) larger area detectors and 4) multiple signal measurements obtained simultaneously while x-ray mapping [9].

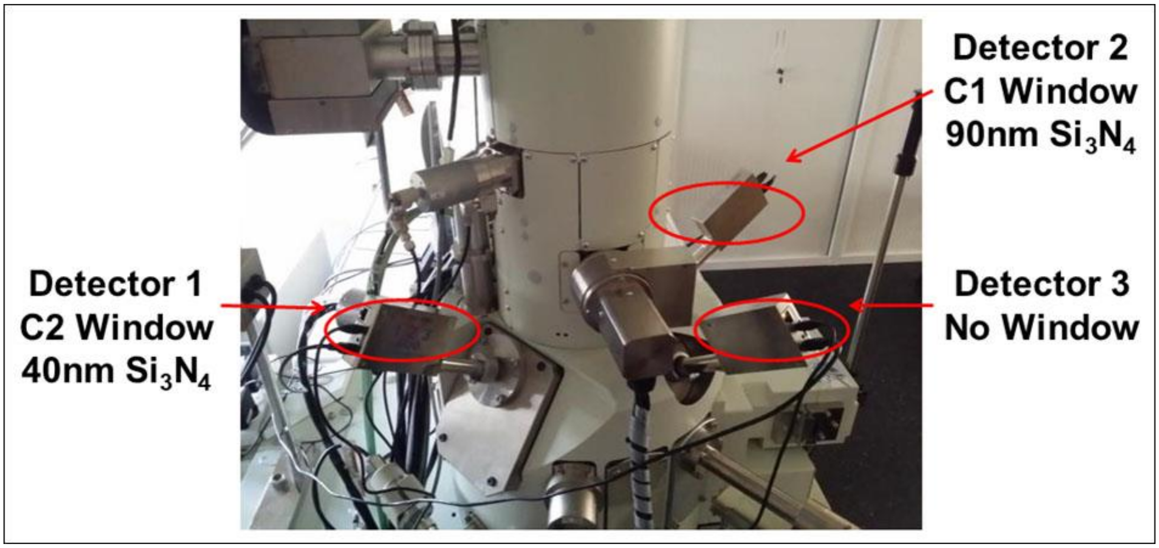
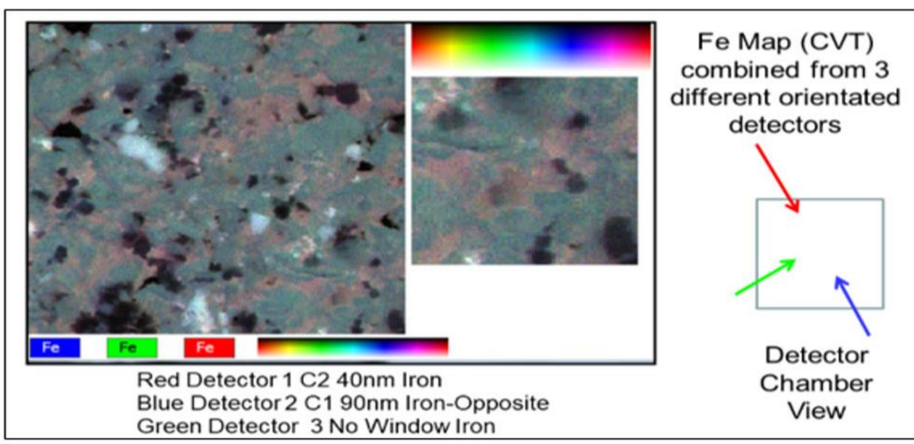
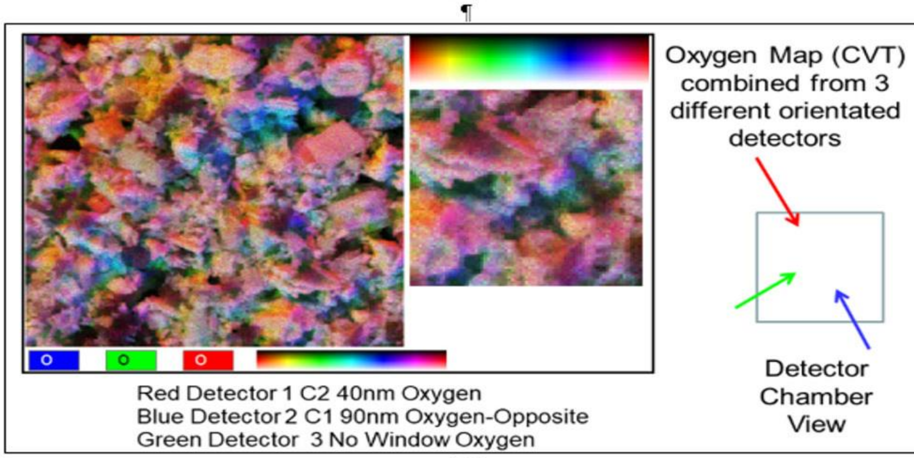



**Figure 1.** JEOL 7001F FEGSEM with multiple Amptek SDD's operating with a Moran Scientific Microanalysis and X-ray Mapping system.




a.




b.

https://doi.org/10.1017/S1431927620013264 Published online by Cambridge University Press

**Figure 2.** Colouring Verification Technique (CVT) where a different red, green and blue (RGB) colour is assigned to each detector for the same element on a Lithium battery electrode. The RGB image a) Fe shows a nearly grey scale map indicating total correlation between the three detectors at the most critical final stage of quantification mainly due to the minimal absorption of Fe Kalpha X-Rays for the current electron penetration. Any colour present is due to variation from different detector orientation. a) Fe map results and b) Oxygen map results show oxygen in direct proportion to the facet facing the respective detector.

References

1. R. Wuhrer and K. Moran, “FEGSEM Dedicated X-Ray Mapping System with Multiple Silicon Drift Detectors for Quantitative X-Ray Mapping”, AMAS2019, 15th Biennial Australian Microbeam Analysis Symposium, 11-15 February Melbourne Victoria, Australia, 2019.
2. R. Wuhrer and K. Moran, “Dedicated X-Ray Mapping System with Single and Multiple SDD Detectors for Quantitative X-Ray Mapping and Data Processing”, Microsc. Microanal. 21 (Suppl 3), 2191-2192 (2015).
3. K. Moran and R. Wuhrer, “Quantitative Bulk and Trace Element X-Ray Mapping Using Multiple Detectors”, Mikrochimica Acta, Vol. 155, pp. 59-66 (2006).
4. K. Moran and R. Wuhrer, “X-ray Mapping and Interpretation of Scatter Diagrams”, Mikrochimica Vol. 155, pp. 209-217 (2006).
5. R. Wuhrer, K. Moran and M. R. Phillips, “Multi-Detector X-Ray Mapping and Generation of Correction Factor Images for Problem Solving“, Microscopy and Microanalysis, 14(suppl 2), 1108CD-1109CD (2008).
6. R. Wuhrer, K. Moran and M. R. Phillips, “X-Ray Mapping and Post Processing”, Microscopy and Microanalysis, 12 (suppl 2), 1404CD-1405CD (2006).
7. R. Wuhrer and K. Moran, “X-ray Mapping Characterisation of Materials that have a Large Dynamic”, Microsc. Microanal. 22 (Suppl 3), 114-115 (2016).
8. R. Wuhrer and K. Moran, “Low Voltage Imaging and X-Ray Microanalysis in the SEM: Challenges and Opportunities”, IOP Conference Series: Materials Science and Engineering Volume 109, Issue 1, (2016).
9. Authors wish to thank Amptek Inc for the detectors, especially John Pantazis and David Clifford. Also a special thank you to Scott Cheevers for machining specialised flanges for the SEM and Western Sydney University Advanced Materials Characterisation Facility (AMCF) and staff.