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An accessible Secondary Electron Hyperspectral Imaging approach to draw meaningful insights from scanning electron microscopy

James Nohl^{1,2}, Jingqiong Zhang¹, Lyudmila Mihaylova¹, Serena Cussen³, Cornelia Rodenburg¹

¹Department of Materials Science and Engineering, The University of Sheffield, Mappin Street, Sheffield, UK, ²The Faraday Institution, Quad One, Becquerel Avenue, Harwell Campus, Didcot, UK, ³School of Chemistry, University College Dublin, Belfield, Dublin 4, Ireland

Background

Hyperspectral imaging in the scanning electron microscope (SEM) commonly uses the energy dispersive X-ray (EDS/EDX) signal. The colour maps produced have become a staple part of analysis for visualising elemental heterogeneity and quantifying bulk elemental composition of hard materials. Now, automated collection and analysis routines have extended to secondary electron hyperspectral imaging (SEHI) and the ability to produce colour maps of surface chemical heterogeneity down to the nanoscale [1].

SEHI, combined with an accessible and automated data analysis tool, has yielded maps of surface chemical bonding, such as the surface functionalisation of soft materials, compounds of light elements, and crystallinity by cross-linking. As well as offering complimentary information to elemental composition, the emission characteristics of SEs offer improved surface sensitivity and spatial localisation versus X-ray imaging in SEM, particularly for softer materials.

Methods

In this implementation, SEHI data volumes are built by sequentially imaging with the through-lens-detector (TLD) with a mirror electrode which steps through a series applied voltage biases to act as a variable low-pass energy filter. Automated data collection routines control the cutoff energy of the low pass filter to acquire images quickly. Imaging is done at 1 kV accelerating voltage and < 50pA beam current to produce suitable signal to noise and SE yield close to 1, as well as to reduce SE2 background emissions, and to limit the beam dose received by the material.

The data volume is differentiated with respect to energy to plot local SE spectra and slices of energy ranges (Figure 1a) are summed to create maps which relate a colour to a chemical feature in the SE spectrum (Figure 1b-d). The energy ranges for each colour channel can be identified without prior knowledge by a blind-source separation using a non-negative matrix factorisation (NNMF) method. The program for creating colour-SEHI images has been packaged and redistributed as a portable MATLAB application.

Results

The first demonstration of the colouring method is on a lithium metal anode used in lithium metal, lithium-sulphur and lithium-air batteries. The surface 'platelets' are heterogenous features in red which measure 250 nm across. Whereas lithium oxide morphologies were imaged over a 25 μm horizontal field width by windowless EDS. Ranking of surface chemical species by comparison to theoretical models was used to identify contributions from lithium hydroxide, nitride and carbon containing species and found the red regions to have a higher proportion of lithium nitrides and hydroxides while the blue range included more lithium-carbon compounds.

Applying the analysis to a spider dragline silk fibre reveals domains of nanoscale ordering due to cross-linking, which measure 45 nm across (coloured green, Figure1c). A higher proportion of cross-linking is indicated by more intense SE emissions at 1.1 eV denoting di-sulphide bridging.

At a length scale of 25 μm horizontal field width, the approach is applied to a perovskite solar cell material, which is a mix of organic methylammonium and inorganic lead iodide (ie. hard and soft) material. The automated approach to colouring identifies a two-phase material, to identify regions of lead iodide (PbI₂) within an organic-inorganic perovskite.

Conclusions

This advance in SEHI analysis is an important extension of LV-SEM capability to map and identify regions of surface chemical bonding. This is achieved through an accessible data collection and analysis routine that applies hyperspectral imaging to the SE signal.

Characterisation of surface chemical bonding heterogeneity on bulk functional materials becomes accessible down to nanoscale even for soft matter, as demonstrated by mapping 45 nm wide cross-linked regions in spider silk where di-sulphide bridges are more prevalent. Light element compound distribution is identified on a lithium metal anode material as well as phase separation in an organic-inorganic perovskite solar cell material.

Having demonstrated the technique can be applied to characterise both organic and inorganic material, future studies will include the lithium metal anode post electrochemical cycling and growth of the solid-electrolyte interphase layer.

Graphic:

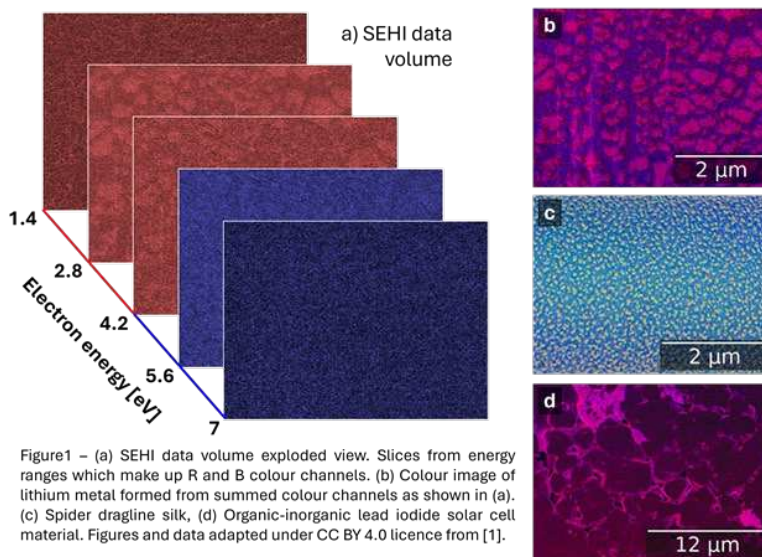


Figure1 – (a) SEHI data volume exploded view. Slices from energy ranges which make up R and B colour channels. (b) Colour image of lithium metal formed from summed colour channels as shown in (a). (c) Spider dragline silk, (d) Organic-inorganic lead iodide solar cell material. Figures and data adapted under CC BY 4.0 licence from [1].

Keywords:

SEHI, SEM, surface chemistry, nanoscale

Reference:

[1] J.F. Nohl, N.T.H. Farr, Y. Sun, G.M. Hughes, N. Stehling, J. Zhang, F. Longman, G. Ives, Z. Pokorná, F. Mika, V. Kumar, L. Mihaylova, C. Holland, S.A. Cussen, C. Rodenburg, *Materials Today Advances*, 19, 2023, 100413, DOI:10.1016/j.mtadv.2023.100413.