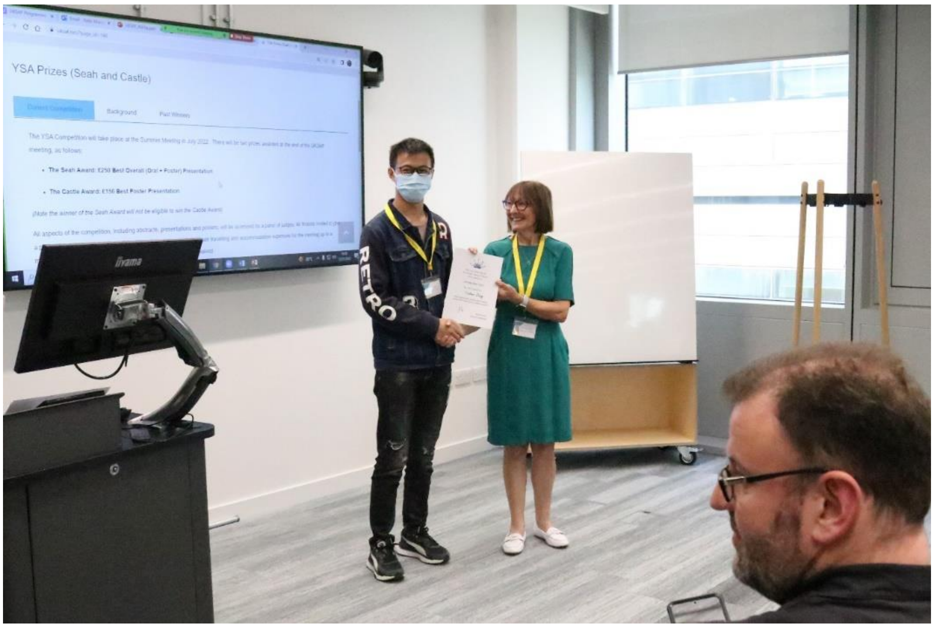
IoP Materials and Characterisation Group

Crossley Award, UK Surface Analysis Forum

2022 (Inaugural)

Yizhuo Ding, The University of Manchester



Yizhou Ding and Alison Crossley at the Royce at the University of Manchester.

Characterisation of Aluminium-lithium alloys using NanoSIMS and EPMA

Yizhuo Ding[1], Kexue Li[1], Joseph Robson[1], Katie L. Moore[1]

[1]The University of Manchester, Oxford Road, Manchester, M21 0XT

Aluminium-lithium alloys are extensively used in aerospace applications due to the improved properties the addition of Li has on the Al alloy system. Low density alloys are extremely important in aerospace applications due to the increasing demand to reduce fuel consumption thereby reducing greenhouse gas emissions and save cost. For every 1 at% of Li added to the aluminium alloy, the density is reduced by 3%. The distribution of Li and its precipitates have a major effect on the properties of the alloy. Although Al-Li alloys have been in development since the 1920's, there are still challenges associated with their production, for example when Al-Li alloys are cast the Li does not remain evenly distributed throughout the casting due to its low density. Current research is investigating if additive manufacturing can generate a uniform distribution of Li in these alloys. However, it is analytically very challenging to spatially localise the Li distribution with traditional techniques such as with energy dispersive X-ray spectroscopy in a scanning electron microscope. As it is difficult to determine the Li distribution it is hard to understand the role it plays in alloy strengthening in conventionally produced and additively manufactured alloys.

In this project high spatial resolution secondary ion mass spectrometry (NanoSIMS 50L) and Electron Probe Microanalysis (EPMA) with a wavelength-dispersive soft X-ray emission spectrometer (WD-SXES) are used to characterise Wire + Arc additive manufacturing (WAAM) produced Al-Li alloys. The results show that in the as-produced alloy the precipitates are highly complex containing a wide range of elements that have co-precipitated. This presentation will show how the NanoSIMS is able to map Li at high lateral resolution which is necessary as the Li-containing precipitates are less than a micron in size. However, the exact type and composition of the complex precipitates are yet to be determined and further complementary EPMA work is required to achieve this. The next stage of this project is to combine the NanoSIMS and EPMA WD-SXES data to quantify the Li in both the precipitates and matrix.

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Mohammed Khan, The University of Nottingham

Mohammed Khan with David Scurr, UKSAF Chair (right) at the University of Leeds.

Understanding the Skin Permeation of Agrochemicals using 3D OrbiSIMS

Mohammed Khan[a], Conor Whitehouse[b], Tim Powell[b], Clive Roberts[a], David Scurr[a]
[a] University of Nottingham,
[b] Syngenta, Jealott's Hill, Bracknell

Over the past decade a surge in agrochemical usage has been witnessed worldwide, owing to advancements in agricultural technology [1], demonstrating improvements in crop quality, productivity and intensification among other benefits. Dermal absorption [2] is a potential route of exposure to agrochemicals with potential adverse health effects [3]. All products must comply with strict health and safety regulations and undergo extensive testing prior to launch.

Aims:

1. Explore the native chemistry of the skin specifically the *stratum corneum*.
2. To understand the routes of skin permeation.
3. The effects of co-formulants on the skin permeation of the active ingredient.

3D OrbiSIMS provided label-free and non-invasive imaging of exogenous and endogenous compounds through skin and leaf with high mass and spatial resolution. The chemical composition of skin and wheat leaf was probed with depth profiling showing variations in ion intensities for different layers as a function of depth. 3D OrbiSIMS achieved the first *in situ* tracking of an agrochemical formulation within skin and leaf including all co-formulants.

[1] M. Tariq *et al.*, *Environ. Int.*, 33, 2007
[2] C.A. Damalas *et al.*, *Toxics.,* 8, 2016
[3] C. Gasnier *et al., Toxicol. Rep.,* 3, 2009

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Xiao Yuan L. Wang, The University of Nottingham

Xiao Yuan L. Wang (centre) with UKSAF Chair David Morgan (left) and Ben Spencer (right) at the University of Surrey.

Native state 3D chemical imaging of polyurea grease using cryo-SIMS imaging and depth profiling

Xiao Yuan L. Wang[1], Davide S.A. De Focatiis[1], David J. Scurr[2], and Derek J. Irvine[1]
[1]University of Nottingham, Faculty of Engineering, UK.
[2]University of Nottingham, School of Pharmacy, UK.

Polyurea thickened greases exhibit high operating temperatures and oxidation stability and hence play an important role in reducing excess friction in machine components. They are complex materials consisting of a base oil and polyurea thickener[1], the latter forms a complex 3D network that traps the base oil molecules in different ways[2]. This structure formed by the thickener is closely linked to the behaviour of the material[1]. Therefore, comprehending their structure provides crucial insight in improving their lubricating performance[3].

Scanning electron microscopy (SEM) is typically used to define the morphology of polyurea grease. However, removal of the base oil is required prior to analysis, which distorts the true structure[3]. Cryogenic SEM (cryo-SEM) is an alternative to image polyurea grease without removing the base oil but suffers from poor contrast between the thickener and base oil components. Manufacture of polyurea greases can also be difficult due to the health hazards of raw materials.

In this study, we explore the possibility of using cryogenic secondary ion mass spectrometry (cryo-SIMS) to characterize the structure of polyurea greases in greater detail than cryo-SEM alone, using a similar protocol employed in the cryo-OrbiSIMS analysis of frozen hydrated biofilms[4]. The analysis was conducted on a commercially sourced polyurea grease sample, with the potential of expanding into materials made with less hazardous chemistries. Polarised optical microscopy and cryo-SEM suggested that the polyurea thickener component of this commercial sample existed in the form of spherical agglomerates. Cryo-SIMS imaging of the same sample reached the same conclusion as spherical shaped distributions of ions corresponding to polyurea were observed. This was further backed up by Raman microscopy, where features of similar shape and size (~20μm) were visible. Additionally, cryo-SIMS depth profiling confirmed that the spherical features observed in the 2D imaging also existed below the surface and were therefore unlikely to be surface contaminants. This novel way of characterising polyurea grease helps improve our understanding of the thickener shape and size needed for optimal performance, and to understand and compare the structure-performance relationship of future lubricants made from safer starting materials.

1. Muller, D. *et al.* Tribology International, **2017**, 110, 278-290.
2. Delgado, M.A. *et al.* Chemical Engineering Research and Design, **2005**, 83(9), 1085-1092
3. Thorseth, M.A. *et al.* Microscopy and Microanalysis, **2021**, 27(1), 12–19.
4. Zhang, J. *et al.* Analytical Chemistry, **2020**, 92(13), 9008– 9015.