Experiment Brief

OnPoint Detector and Octane Elite Super EDS System

Title

Quantitative mapping of lithium in a scanning electron microscope

Instruments Used

We bring together Gatan's OnPoint™ detector and EDAX's Octane Elite Super EDS system to map lithium in the scanning electron microscope (SEM).

Background

Lithium-containing compounds and alloys are critical in our efforts to meet energy reduction commitments. Undoubtedly, we have seen enormous strides in the development of these materials but perhaps, what is even more impressive is that this has largely been achieved without a robust method to reveal or quantify the distribution of lithium at the microscale. A quantitative method to map lithium in bulk materials remains one of the holy grails for microanalysts as energy dispersive x-ray spectroscopy (EDS) is largely ineffective for elements with an atomic number lower than 4. However, quantification of Li in the SEM was demonstrated recently using a composition by difference method based on EDS and quantitative backscattered electron imaging (qBEI) [J.A. Österreicher et al., Scripta Materialia 194 (2021) 113664]. We extend the composition by difference method to generate quantitative, spatially resolved elemental maps of a MgLiAl alloy.

Materials and Methods

The composition by difference method uses EDS to quantify elements Z = 4 – 94, while qBEI determines the mean atomic mass (the qBEI signal being a function of atomic number for Z = 1 – 94). The fraction of 'missing' light elements (Z = 1 – 3) is then calculated based on the difference in the two data sets. A MgLiAl was prepared using the Ilion® broad beam argon ion polisher and analyzed in a field emission SEM using the OnPoint backscattered electron (BSE) detector and Octane Elite Super EDS system. Quantification of the BSE data and calculation of aluminum, magnesium and, for the first time, lithium elemental maps was performed in DigitalMicrograph® software.

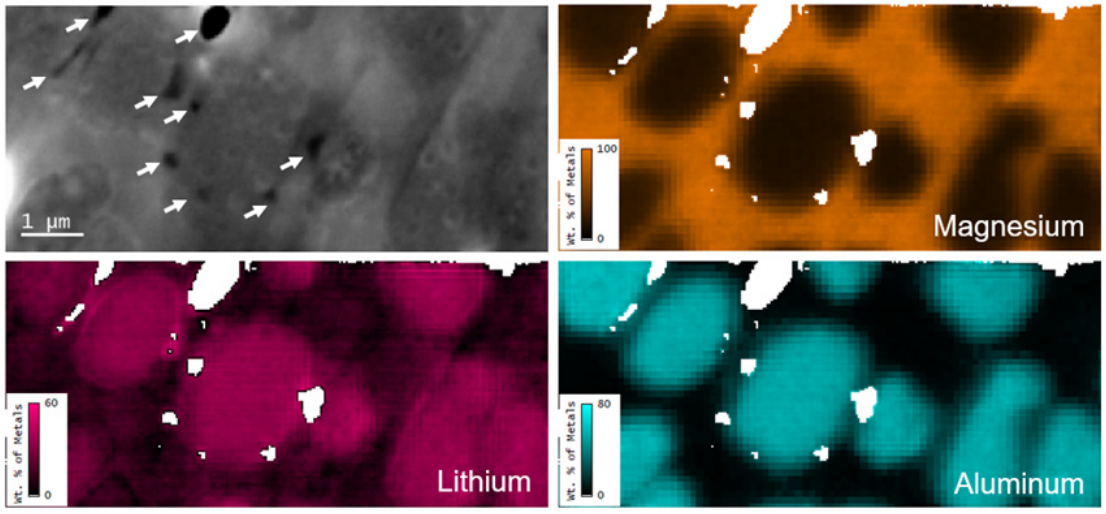

Figure 1. Secondary electron image and elemental metal fraction maps (by wt. %) of the same region of the MgLiAl alloy; white pixels are regions excluded from the analysis due to influence of topography (identified by arrows in the secondary electron image) shown here for clarity.

Summary

Single-digit mass percentages of Li were mapped quantitatively in the SEM over a field of view of tens of microns with sub-micron spatial resolution. The lower detection limit and insensitivity to the chemical bonding state make this a very attractive technique for spatially resolved analysis of low atomic number elements.

Credit(s)

A special thanks to Johannes Österreicher and colleagues, Austrian Institute of Technology for providing the sample and invention of the composition by difference method

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