doi:10.1017/S1431927622001258
Microsc. Microanal. 28 (Suppl 1), 2022

Plasma FIB-SEM-based Kintsugi Imaging of Battery Electrodes

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The mesostructure of porous electrodes used in lithium-ion batteries strongly influences cell performance [1]. Accurate imaging of the distribution of phases in these electrodes would allow this relationship to be better understood through simulation [2]. However, imaging the nanoscale features in these components is challenging. While scanning electron microscopy is able to achieve the required resolution, it has well established difficulties imaging porous media. This is because the flat imaging planes prepared using focused ion beam milling will intersect with the pores, which makes the images hard to interpret as the inside walls of the pores are observed. It is common to infiltrate porous media with resin prior to imaging to help resolve this issue [3], but both the nanoscale porosity and the chemical similarity of the resins to the battery materials undermine the utility of this approach for most electrodes.

In this study, a technique is demonstrated which uses *in situ* infiltration of platinum to fill the pores and thus enhance their contrast during imaging (Fig. 1). Reminiscent of the Japanese art of repairing cracked ceramics with precious metals, this technique is referred to as the *kintsugi* method. The images resulting from applying this technique to a conventional porous cathode are presented and then segmented using a multi-channel convolutional method [4] (Fig. 2). We show that while some cracks in active material particles were filled with the carbon binder phase, others remained empty, which will have implications for the rate performance of the cell. Energy dispersive X-ray spectroscopy was used to validate the distribution of phases resulting from image analysis (Fig. 2), which also suggested a graded distribution of the binder relative to the carbon additive. The equipment required to use the kintsugi method is commonly available in major research facilities and so we hope that this method will be rapidly adopted to improve the imaging of electrode materials and porous media in general.

References:

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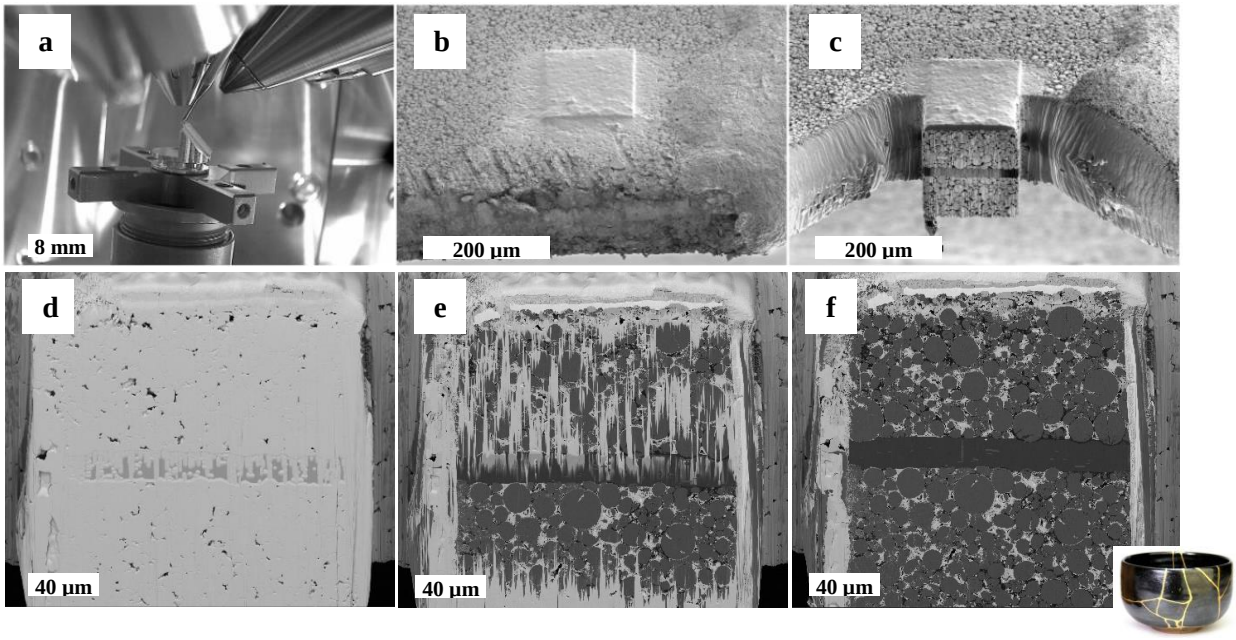

Figure 1. The cross-section preparation workflow using Helios Hydra PFIB-SEM. (a) Image of the sample arrangement relative inside the vacuum chamber. (b) deposition of the Pt-C mix protective layer on the top surface of cathode. (c) the material block and block face preparation. (d) Pt layer deposition on the block face; (e) the automated ASV run, removing the excess of Pt layer. (f) image of the kintsugi infiltrated block face at 54 nm resolution (Inset shows an example of the pot repaired using the traditional kintsugi method). The scalebar in (a) is 8 mm; in (b, c) is 200 µm; and in (d-f) is 40 µm.

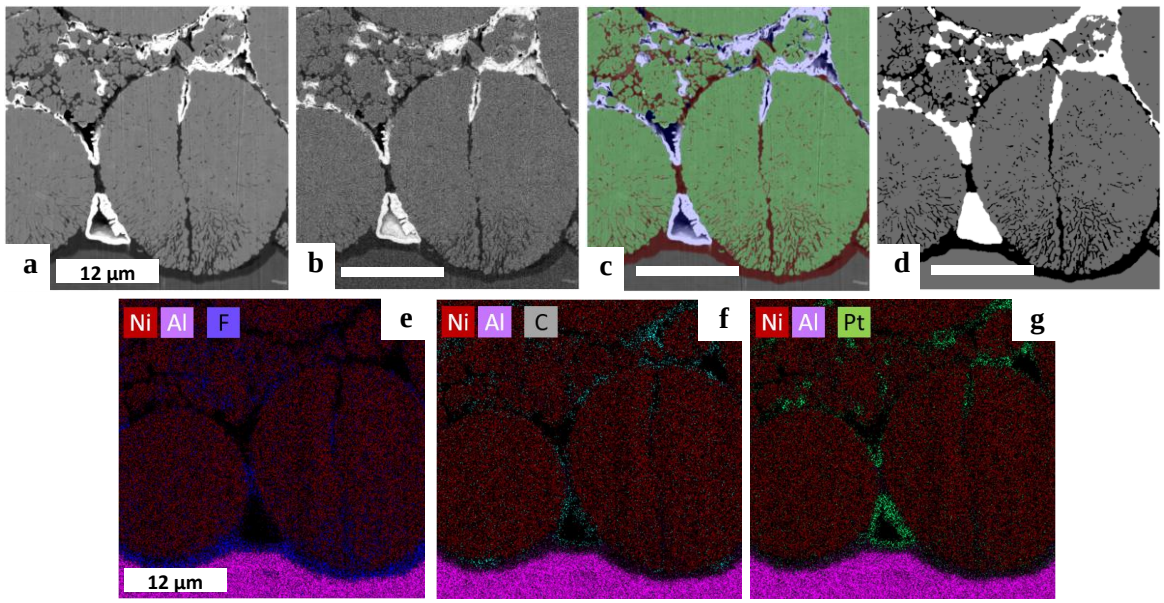

Figure 2. Images of cathode cross-section prepared using kintsugi method. (a) SEM-BSE images using CBS detector. (b) SEM-BSE images using TLD. (c) CBS image overlaid with segmentation false colouring, where green is active material, red is CBD, and blue is pore. (d) Segmentation where white is pore, black is binder and grey is either active material or current collector. (e-g) Composite of EDS images highlighting the distributions of Al (current collector), Ni (AM particles), Pt (pores), and C (CBD), and the presence of F (polyvinylidene fluoride binder).

https://doi.org/10.1017/S1431927622001258 Published online by Cambridge University Press