Protocol for Characterization and Post-test Analysis by SEM, FIB-SEM

The short stack was disassembled from the set-up and embedded in resin at HTc. The short stack was therefore immersed in liquid resin and underwent degassing before reticulation. Mechanical compression of the repeat elements was lost before the embedding. Due to the important size and complexity of the short stack, degassing was not complete and clearly and some channel are areas were not completely infiltrated by the epoxy resin.

Polishing process was the same as for small coupons, a medium portion of the embedded stack was first cut by an industrial saw and later the smaller portion to be polished was cut via diamond disc. This sample was then manually polishing going through these different SiC papers: 320 µm, 600 µm, 1200 µm, 2500 µm, 4000 µm and finally perfected with diamond paste: 6 µm, 3µm and 1 µm. Finally, polished cross sections were carbon coated to avoid electrostatic charges during SEM observations.

Post-test analysis were done by SEM observations coupled with quantitative and qualitative EDS chemical analysis. Notably, SEM is a FEI TENEO while EDS detector is provided by Bruker. Typical beam voltages were 15 kV and 30 kV while working distance was kept between 10 and 8 mm. The error of the EDS quantification is taken as 1%. Most of the pictures were taken in backscattered electron mode to highlight difference in elemental composition.

Image post treatment was done coupling Adobe Photoshop, ImageJ and matlab. Data processing was done via Microsoft office Excel and Matlab. The software to treat chemical analysis is provided by Bruker.

Sample preparation for electron microscopy/quantitative analyses (SEM, FIB-SEM)

Pristine and aged cell samples were provided by SOLIDpower for quantitative 2-D and 3-D analyses. The received chunks of material were first fractured to expose the interface between the anode and the YSZ electrolyte and impregnated using EPON812. Pre-impregnation was performed by progressively increasing the resin/acetone ratio up to one in typically 7 levels, which exceeds the standard datasheet procedure. The samples were polished first mechanically, then by ion-milling for preliminary 2-D SEM observations. The region close to the interface with the electrolyte was then imaged by FIB-SEM serial sectioning. Before the acquisition, fiducial marks were milled on deposited carbon and Pt layers to adjust the position of the FIB beam and maintain the thickness variation of the slices below the nanometer level. The acceleration voltage was of 1.7-1.8 kV and data from both the energy selective backscatter (ESB) and in-lens secondary electron detectors (or alternatively secondary electron/secondary ion detector), were recorded. The pixel size and slice thickness were respectively set to obtain isometric voxels, typically of 7-15 nm (10 nm for most reconstructions).