Detection and identification of iron and steel particulate in 4200 hour Bus stack MEAs using SEM and TOF-SIMS

M.L. Bos.

Failure Analysis group, R&D, Ballard Power Systems Inc.,
9000 Glenlyon Parkway,
Burnaby, B.C., Canada, V5J 5J9.

One of Ballard Power Systems Inc. business lines is striving to replace diesel engines in commercial buses with fuel cell powered engines, also known as stacks. Fuel cell engines will reduce greenhouse gas emissions by eliminating the need to use fossil fuels; hydrogen gas is typically used as the fuel. Ballard currently has 26 fuel cell powered buses in commercial operation in Hamburg, Germany and Whistler, B.C., Canada. Bus fuel cell engine durability is a significant issue in order to keep warranty costs down. Failure analysis is a key function in understanding failure modes and provides engineers with the insight to improve fuel cell durability.

The heart of the fuel cell stack is the MEA (membrane electrode assembly). The Bus fuel cell stack under investigation ran with two 204-cell Mark 1100 MEA cell rows. The stack was slated to run 6000 hours in its system module packaging, however, at about 4000 hours, the stack internal reactant leak rate began to increase. At 4200 hours, the test was stopped and failure analysis commenced to determine the cause of the leaks.

Examination of the anode fuel supply circuit identified loose metallic particulates both on the stainless steel 316 inlet adaptor and plastic supply hoses. Energy Dispersive x-ray Spectroscopy (EDS) analysis of the particulates detected the presence of aluminum, iron, copper, and traces of magnesium, potassium, sodium, and sulfur. Chromium and nickel, the main components in stainless steel 316, aside from iron, were not detected.

Low levels of iron contamination in MEAs have been shown to initiate radical attack of ionomer membrane in the MEA, leading to internal transfers [1, 2]. Therefore it was important to identify whether the failure mode was related to iron and steel contamination or some other failure mode.

Cross-sectional analysis of the MEAs by Scanning Electron Microscopy (SEM) with EDS analysis did not detect any steel and iron particles, although this was likely due to these metals being below the EDS detection limit (~0.1 atomic percent). The same epoxy-potted samples were ion-etched to remove polishing residue and examined by Time-Of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS). TOF-SIMS was able to identify steel particles in the MEA gas diffusion layer as well as imbedded in the membrane, in the vicinity of failures that were initiating [3].

References

[1] T. Kinumoto, et al., *J. Power Sources, 158* (2006) 1222.
[2] A.B. LaConti, et al., Technical memo, 68-2, General Electric Company (1968).
[3] The author would like to thank Jim Gibson of Evans Analytical Inc. for performing the TOF-SIMS analysis, and Jielin Song for doing the sample preparation.

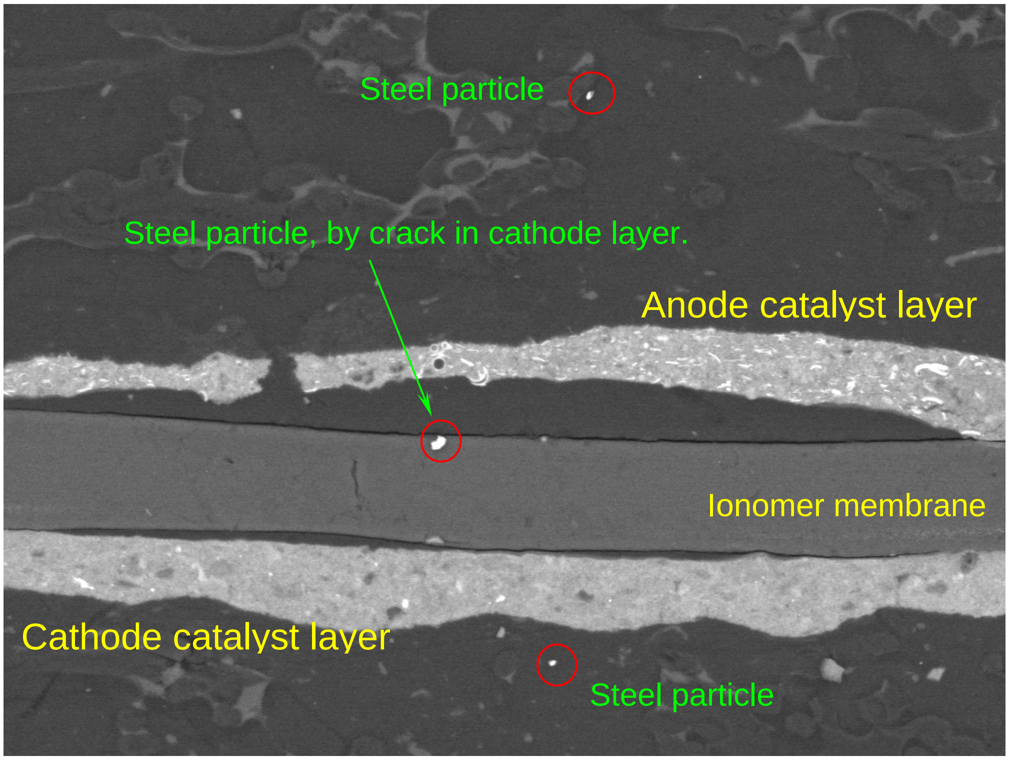

Figure. 1. Back-scatter electron detector SEM cross-section of a bus MEA with steel particles in gas diffusion layers and on membrane.