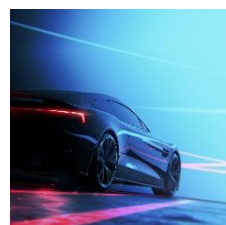


XGT

## Non-destructive failure analysis of a commercial PEMFC using a micro-XRF with a super large chamber



Application Note

Energy XGT36

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**Abstract:** We introduce non-destructive failure analysis on a large membrane electrode assembly (MEA) from a commercial PEMFC stack using a HORIBA XGT-9000SL, a micro-XRF with an extra-large chamber capacity. Thanks to the chamber capacity, we successfully performed non-destructive elemental imaging on a defective area without cutting the sample, and we found unexpected distributions of Fe, Ni, Mn and Cr within the defective area, and that the elements are present in the MEA as a result of possible degradation of the bipolar plate.

**Keywords:** PEMFC, MEA, failure analysis, elemental analysis, micro-XRF

### Introduction

Proton exchange membrane fuel cells (PEMFCs) have been gaining popularity as new clean energy devices for transportation. Failure of a PEMFC is detrimental for passenger safety, so post-mortem failure analysis with analytical instruments is essential to quickly identify the root cause and propose effective counter-measures.

However, conventional analytical instruments often lack sufficient sample chamber capacity to hold the large MEA typical of commercial fuel cells. Usually, large MEAs must be cut into pieces for failure analysis, which is inherently destructive, time-consuming, can introduce contaminants, and negatively impacts further investigations.

In this application note, we will demonstrate the HORIBA XGT-9000SL's ability to both analyze the elemental composition and distribution of a defective area of a large MEA and provide elemental information on how the degradation occurred.

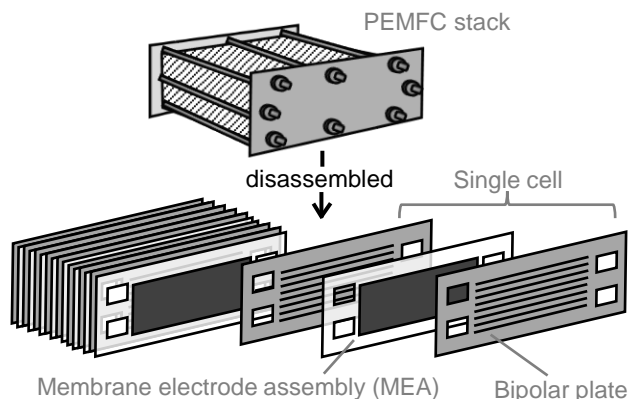


Figure 1. Schematic diagram of a PEMFC stack

### XGT-9000SL

HORIBA's XGT-9000SL is a micro-XRF configured with a super-large chamber, large enough to accommodate an entire MEA from a commercial PEMFC, thereby avoiding the typical need to cut the MEA into pieces. Thanks to the micro-spot size probe and the motorized XYZ stage, the XGT-9000SL can perform high spatial resolution elemental imaging at the microscopic level across an area up to 350 mm x 350 mm. The XGT-9000SL has X-ray shields, and fully complies with the radiation safety requirements outlined in IEC-61010-1. The large capacity and full shielding make it possible to perform non-destructive elemental analysis on a large MEA sample without compromising users' safety.

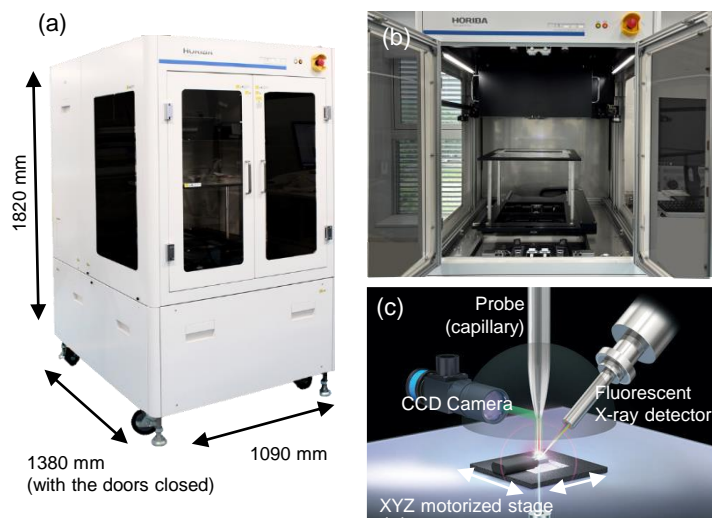


Figure 2. (a) XGT-9000SL (b) the chamber capacity (1030 mm (W) x 950 mm (D) x 500 mm (H)) (c) schematic diagram of the optics.

## Sample information

We obtained a commercial PEMFC stack that had a voltage failure under over-current operation. We disassembled the stack into individual cells and found some visible defects on the surface of the MEA of the cell.

## Measurement & Result

We selected one of the MEAs and performed elemental imaging of the defective area. Figure 3 shows an optical camera image of the defective area and the corresponding distribution images of the elements found. We found Pt, which is an expected key electrocatalyst element, across the entire surface with some areas of significantly reduced signal intensity that correspond with the visible defects. We also detected some unexpected elements, including Fe, Ni, Mn and Cr, within the defective area. To confirm the possible source, we also analyzed a bipolar plate

which is next to the MEA, and found that the Fe, Ni, Mn, and Cr corresponded to the composition of the bipolar plate. This provides evidence that the defect comes from possible degradation of the bipolar plate (the bipolar plate results are not shown here). It is also interesting to note the different spatial distributions of Fe, Ni, Mn, and Cr on the defect region. With further analysis, these results can clearly provide additional information on the degradation mechanism, which allows QC and R&D teams to propose effective counter-measures.

## Conclusion

Thanks to the large sample chamber capacity of the XGT-9000SL, we were able to identify what elements existed, and where, in the defective area without time-consuming sample preparation. This demonstrates HORIBA's XGT-9000SL as an effective failure analysis tool for commercial PEMFCs comprised of large MEA.

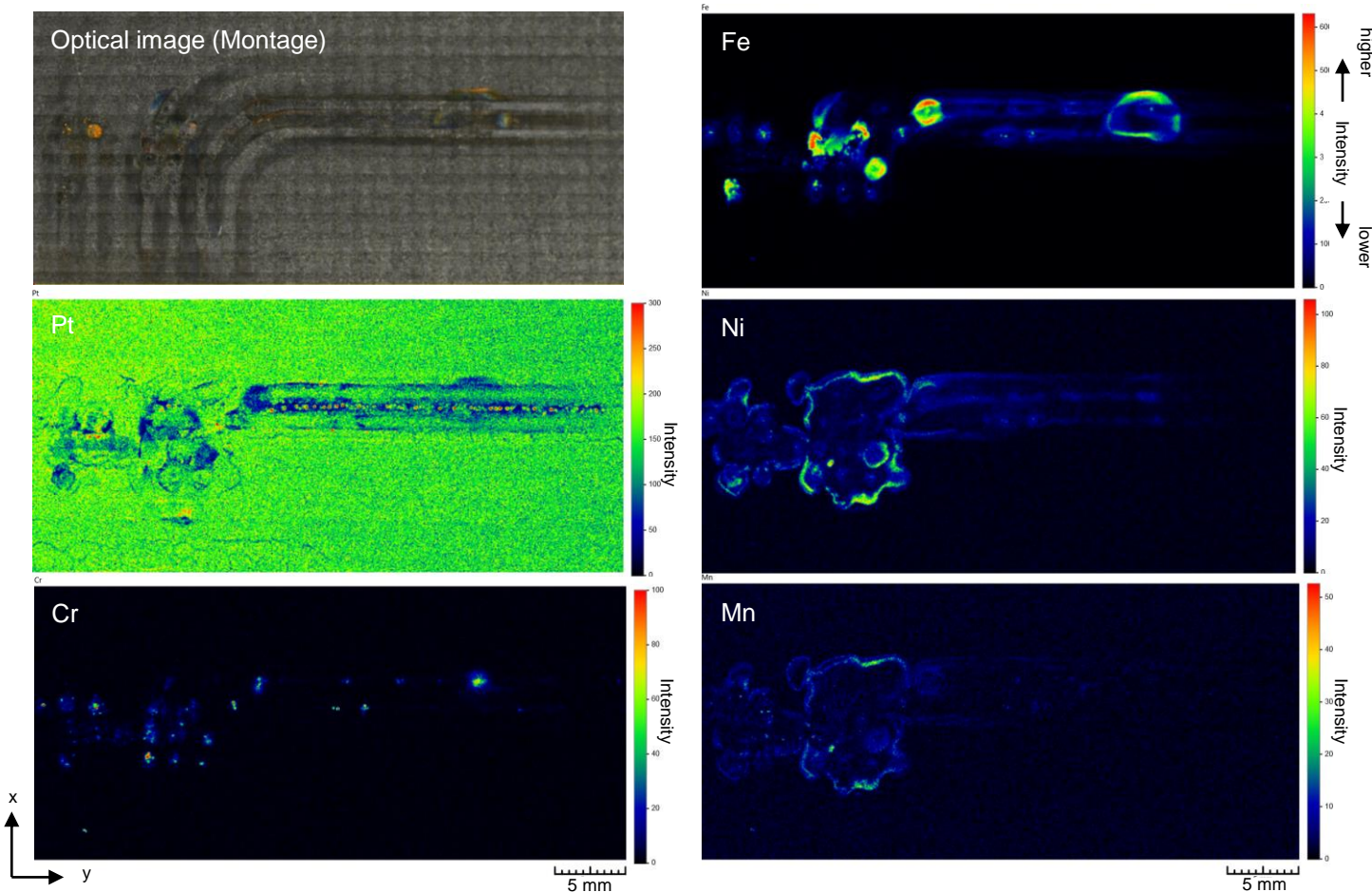


Figure 3. Optical camera image and elemental distribution images of the defective area on a MEA using the HORIBA XGT-9000SL [Mapping condition] XGT-9000SL, 50kV, 600  $\mu$ A, Process 3, Partial vacuum, 40.96 mm x 18.88 mm, Total time 67 min.