

Characterization of Samples Coming from Project Fuel Cells (pristine or operated) by Optical and Electron Microscopy

Aim:

This internal protocol aims at recommending common magnifications for samples characterization by optical and electron microscopy, which ensures easy and comparable analysis of images produced in different laboratories. Every other magnification is also acceptable. However, the magnifications proposed in this protocol are OBLIGATORY.

Introduction:

The methods of contrast mostly used are the BSE (Backscattered electrons, sensitive to local compositional changes), SE (Secondary Electrons, sensitive and more used when the topography of the image, e.g. porosity, is important). The analyses are performed by Energy Dispersive X-ray Spectroscopy (EDXS) calibrated to measure from light elements (e.g. O) up to heavy elements.

SEM operates at 20kV of acceleration voltage, between 2.1 and 2.8 A and high vacuum. If the sample has to be mounted in cross section, then a preparation protocol should be followed: the cell is mounted in an epoxy resin and then polished up to diamond suspension of 0,25 micrometers of grain size. The end of this operation allows the investigator to observe the cross section of the cell by optical microscopy for a first overview.

In order to proceed to the SEM observations and EDXS analyses of the samples, the surface of observation is gilded with a metal coater (sputtering) applying 15nm of gold on the surface.

Mandatory Magnifications:

Electron Microscopy of sample analysis “as received”

500x for a general overview;
1000x for a more detailed observation;
5000X for images useful for image analyses.

Optical microscopy magnifications:

x25;
x100;
x200;
x500;
x1000.

Backscattered electrons magnifications:

200x for the complete overview of the cell (it allows to check the number of layers and to measure their thickness);
1000x for the details;
5000x for the image analysis of the vol. fraction of the phases and for average analysis (the wholeframe is analysed by EDXS in order to have an average composition of an area of 2328 square microns, 58.5 x 39.8 umxum),
x8000 for spot and phase chemical analysis.

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