Microstructural Characterization of PEM Fuel Cells

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Objective

- DOE technical targets: Precious metal loading and cost
  - Use TEM characterization techniques to provide information necessary to optimize the distribution of precious metal catalyst for **increased efficiency and reduced catalyst loading** leading to cost reductions.
- DOE Technical target: durability
  - Characterize/quantify microstructural changes and relation to the **performance loss** in PEMFCs upon use in a fuel cell system to understand issues relating to durability and lifetime.

Approach

- Develop special techniques and procedures to prepare cross-sectional samples suitable for TEM observation from the cathode to the anode, to facilitate the ability to understand the entire cell as a unit. These samples must maintain the spatial relationships among the constituents of the cell in order to achieve insight into the operation of the real system.

Approach

- Characterize the microstructure and chemical composition of PEM cells before and after use to correlate performance to the microstructure down to the nm level.
- Characterize the role of processing in the development of the PEM electrode microstructure: e.g. Nafion content, catalyst particle type, catalyst ink application, etc.

Project Timeline

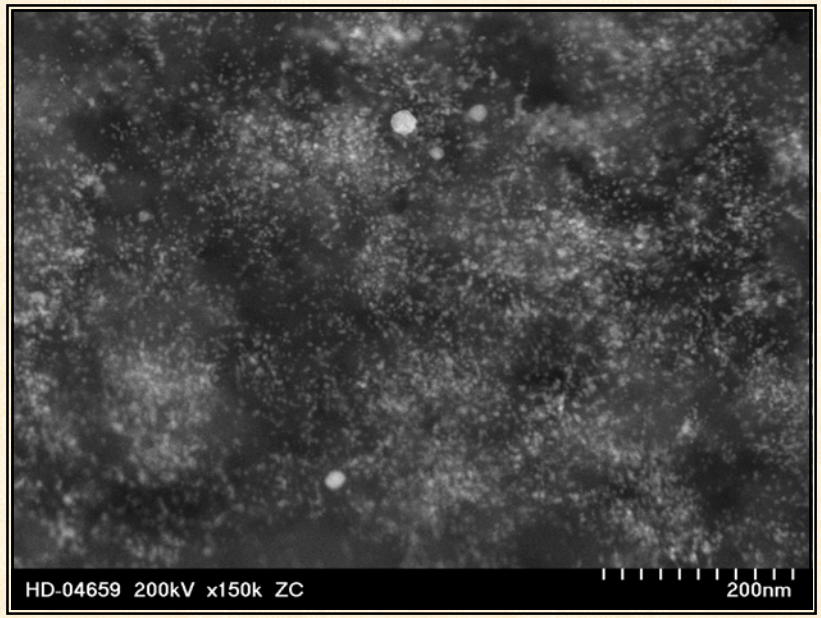
- Start of project **September, 1999**
- First successful cross-section **January, 2000**
- First fresh vs. aged microstructural comparison **June, 2000**
- Pt catalyst size distributions and microchemical changes at PEM/electrode interfaces **June, 2001**
- Establish new cryo-ultramicrotomy facility **October, 2001**
- Successful room-temperature ultramicrotomy **January, 2002**
- Analysis of failed MEA **June, 2002**
- Microstructural changes occurring during MEA assembly (with LANL) **June, 2003**
- Cryo-ultramicrotomy for thinner samples **October, 2003**
- Feedback aging changes to MEA designers **June, 2004**

Highlights for Fiscal Year 2003

- Room temperature ultramicrotomy successful at sub-100 nm thick sections
  - Eases overlap/projection problem
- Secured funding for acquisition of Hitachi HD-2000 STEM
  - Powerful analytical electron microscope well suited for characterization of PEM fuel cell cross-sections
- Examined a number of LANL produced PEM fuel cells to understand the processing-microstructure-performance relations in PEM fuel cells

Thinner is better – advances in room temperature ultramicrotomy

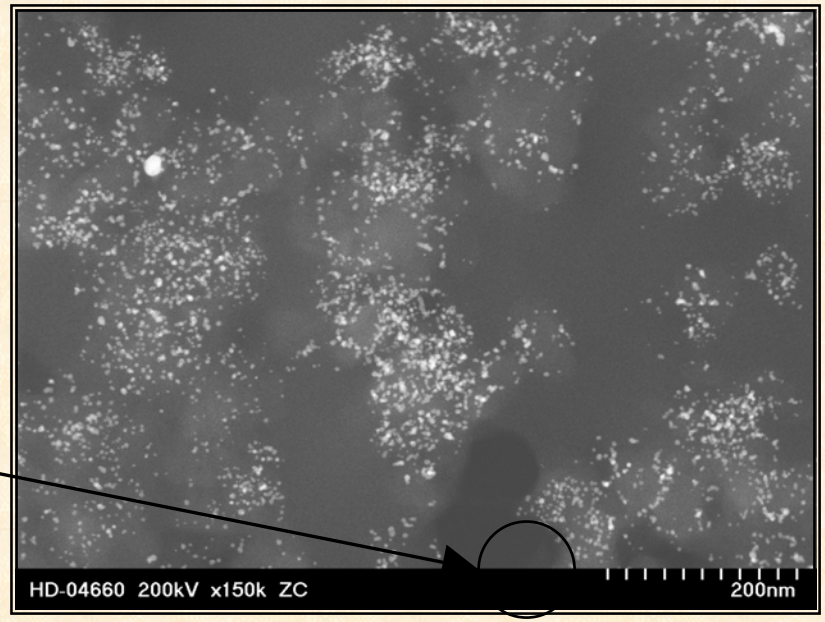
- Initial sectioning produced samples 300 nm thick
- Can see catalyst particles, but not porosity and support phases – catalyst coarsening is NOT the whole story



300 nm

Thinner is better – advances in room temperature ultramicrotomy

- Improved technique provides more information about ALL components
- Porosity visible at bottom
- Catalyst particles and their relation to support visible – 3 phase boundary is where active material lies

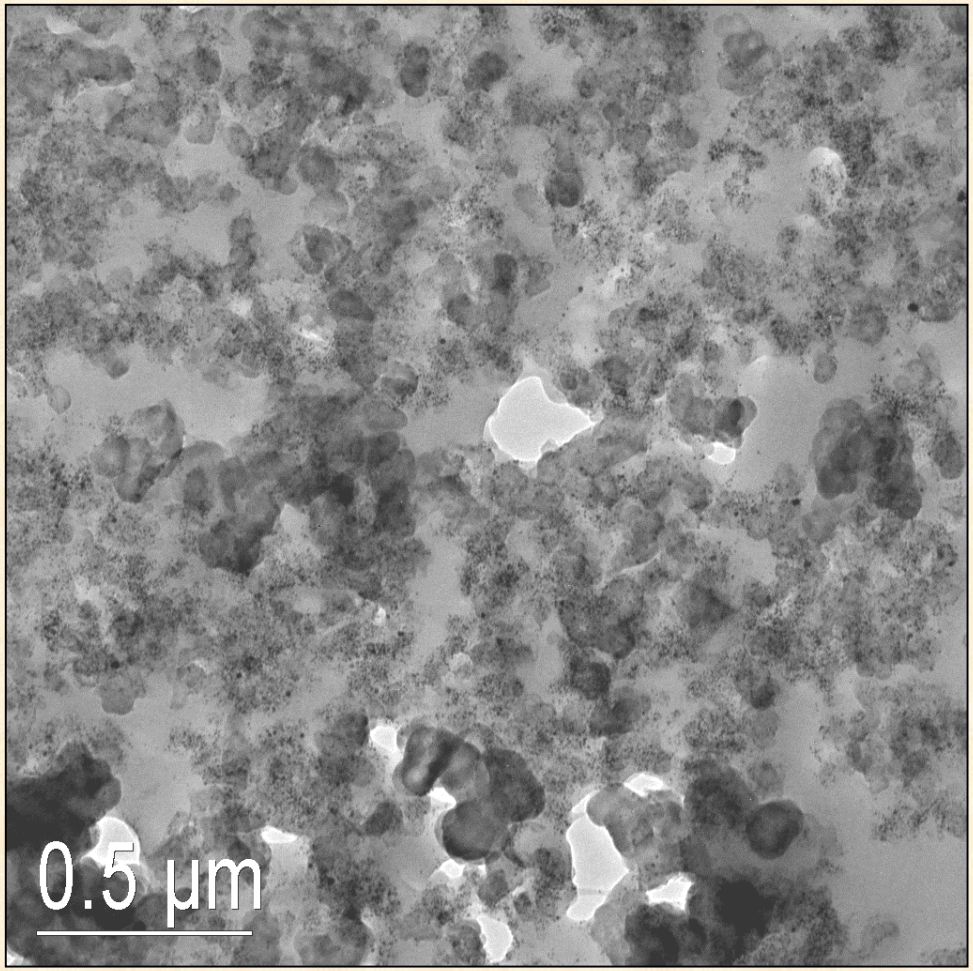


50 nm

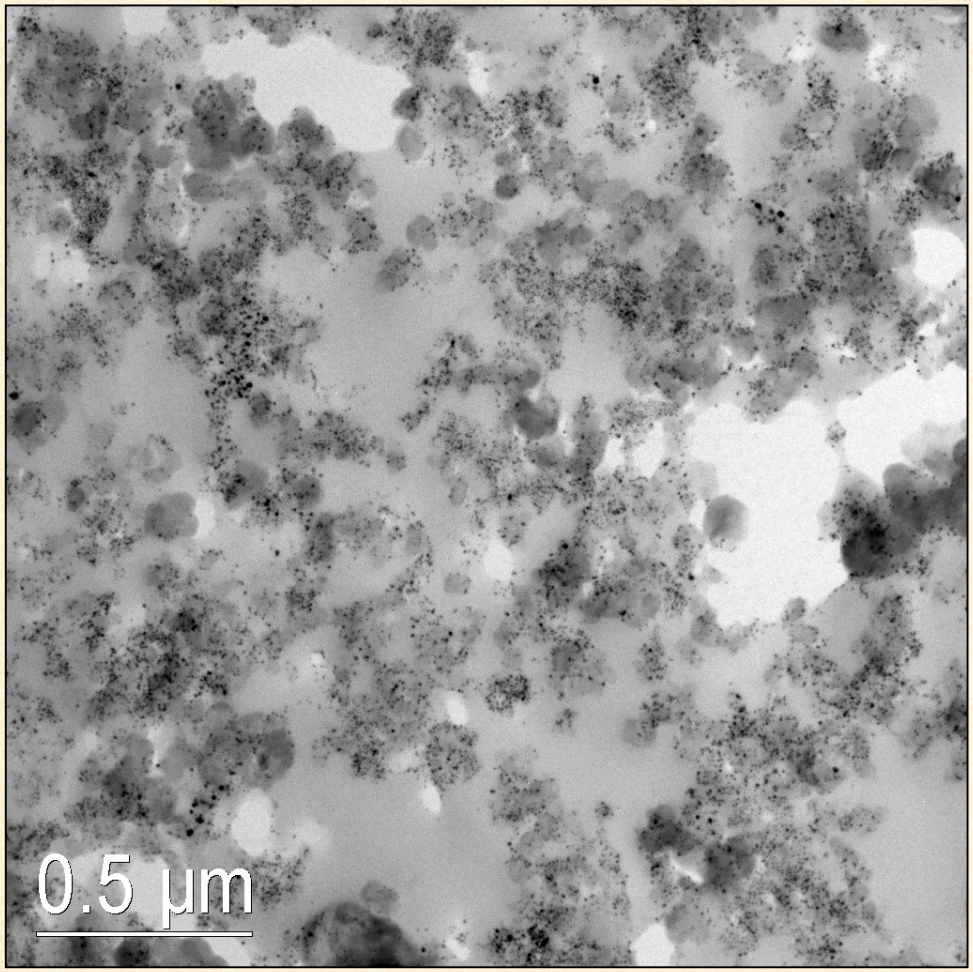
HD-2000 STEM on order

- 0.3 sr solid angle EDX detector – fast, accurate elemental analysis
- Z-contrast detector – ideal for imaging Pt (or other heavy elements) on C
- 2D composition maps easily achieved

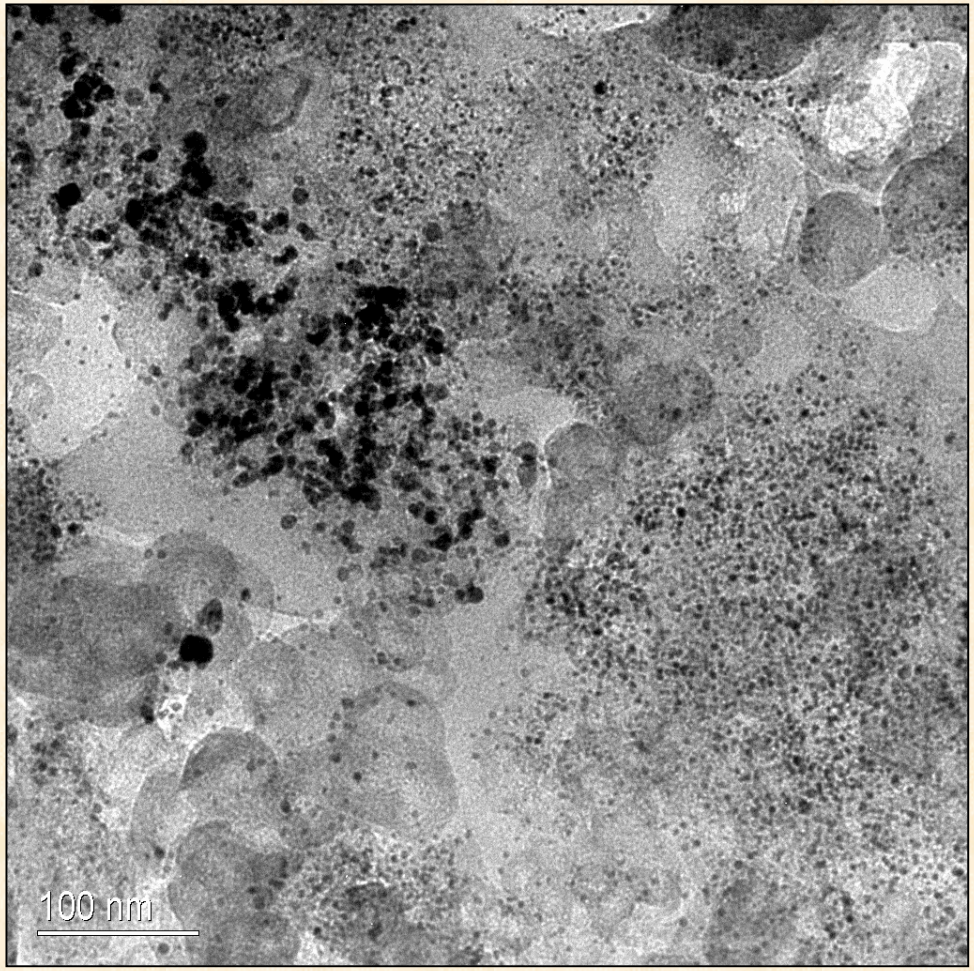
LANL PEMFC without sulfuric acid boiling step



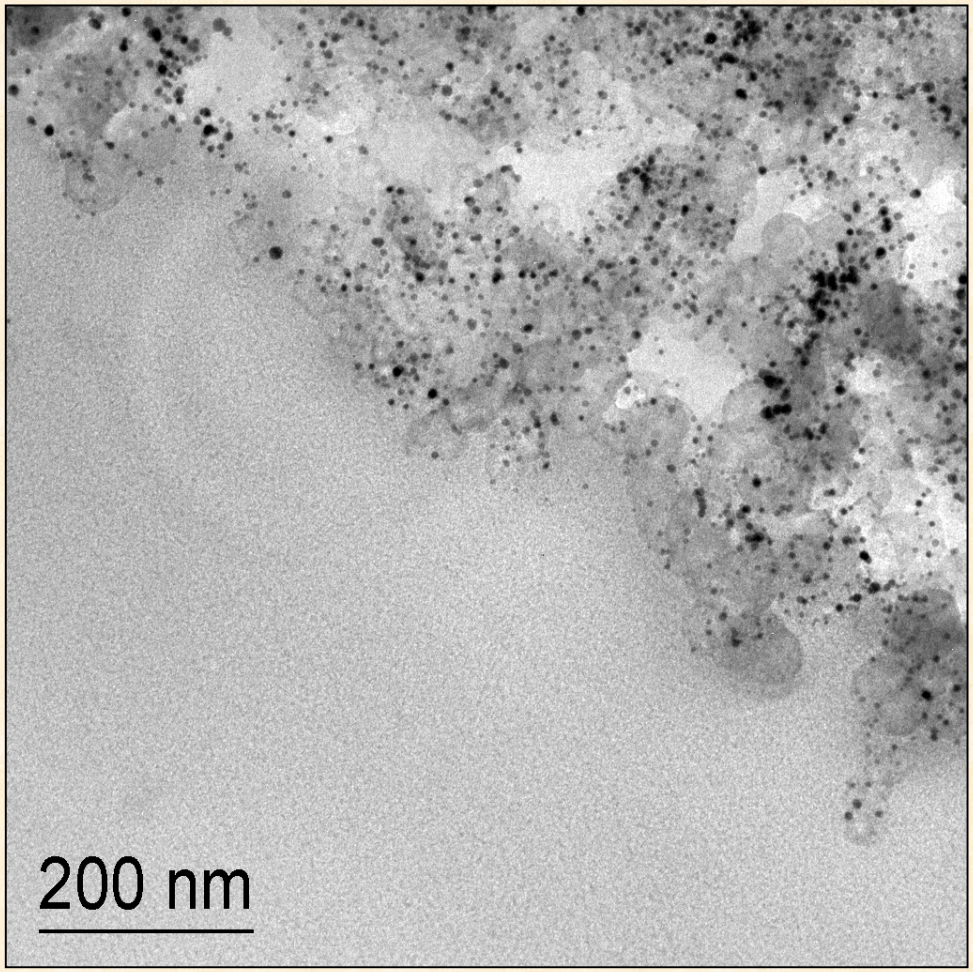
With boiling step



Spatial distribution of particle size distribution for fresh LANL MEA



400 hour life tested LANL MEA



Collaborations

- **LANL providing a series of PEM fuel cells with varying initial processing.**

Plans and Future Milestones

- Improve cryo-ultramicrotomy technique for preparation of ultra-thin (<<100nm) to achieve improved analytical results
- Continue collaboration with LANL on the microstructural changes which occur during the MEA preparation process and report on results

Response to Comments from 2002 Review

- Cyro-ultramicryotomy sample preparation should work to generate <100 nm thick samples – the technique would be valuable if this could become the "routine" sectioning method
  - Work in progress to improve cryo-sectioning capability

Response to Comments from 2002 Review

- More effort to establish more routine EDX data capability if the full advantages for understanding of MEA structural and chemical changes are to be realized
  - New instrumentation acquisition underway to integrate analytical information fully into characterization