

Microstructural Characterization of PEM Fuel Cells

Doug Blom

Larry Allard

ORNL

Objective

- 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.
- Characterize/quantify microstructural changes and relation to the **performance loss** in PEM MEAs upon use in a fuel cell system to understand issues relating to durability and lifetime.

Approach

- Characterize the microstructure and chemical composition of PEM MEAs before and after use to correlate performance to the microstructure down to the nm level.
- Develop special techniques and procedures to prepare samples for TEM observation that are thin over large areas, to facilitate the ability to understand the entire MEA as a unit.
- TEM samples must maintain the relationships among the constituents of the MEA in order to achieve insight into the operation of the real system.

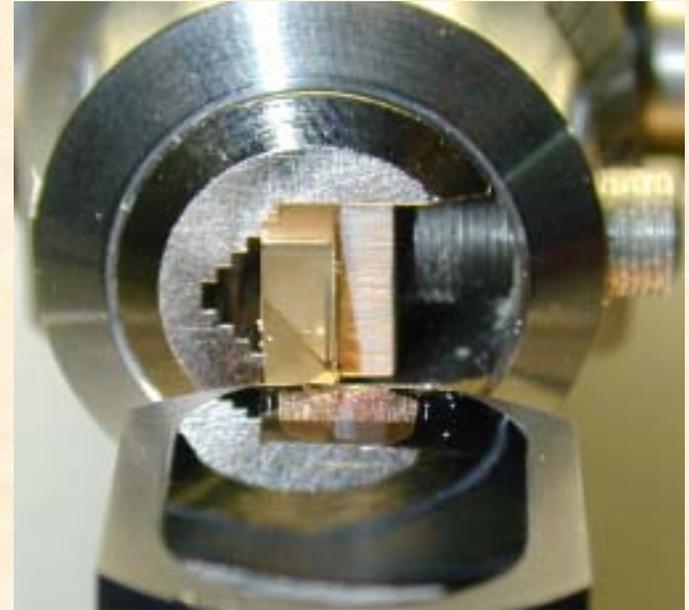
Project Timeline

- 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**
- Cryo-ultramicrotomy for thinner samples **June, 2003**
- Microstructural changes occurring during MEA assembly (with LANL) **June, 2003**
- Feedback aging changes to MEA designers **June, 2004**

Accomplishments This Year

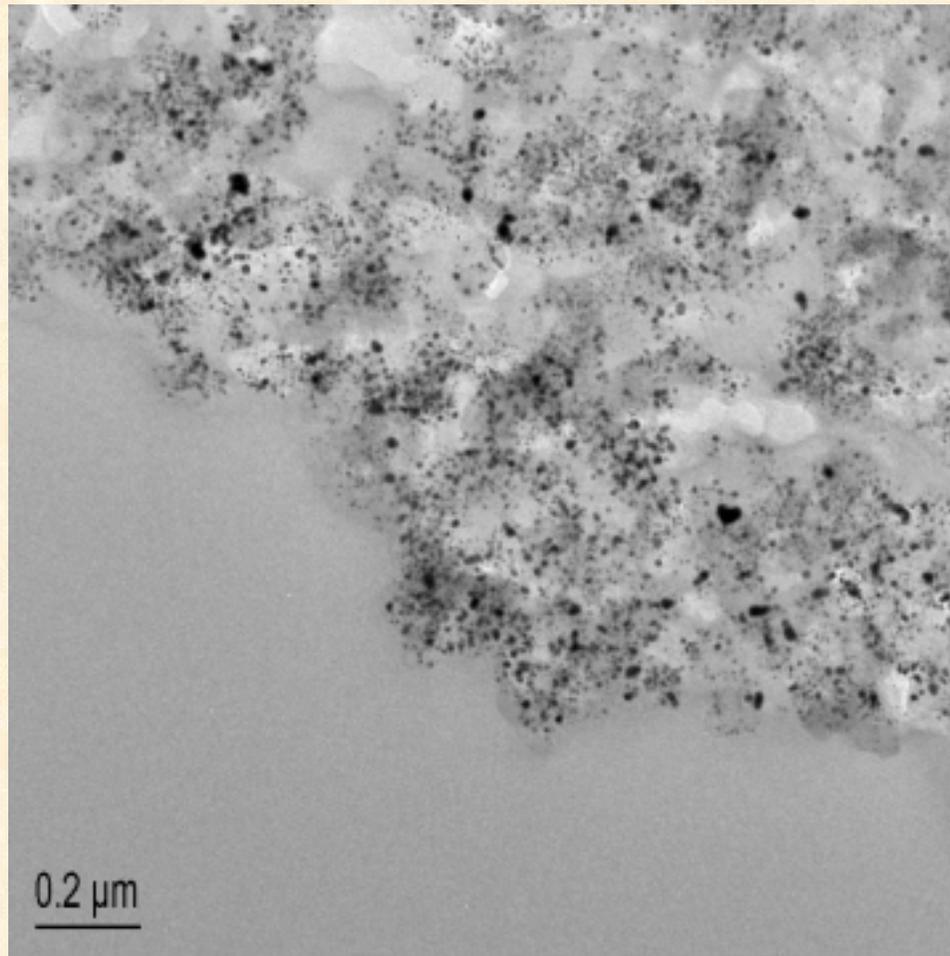
- New state-of-the-art cryo-ultramicrotomy facility established at ORNL
- Prepared successful sections of MEA tested to failure
- Characterized microstructure of failed MEA (aged 1200 hours)
- Quantified change in Pt catalyst size distribution upon aging (fresh vs. short time, vs. failed)

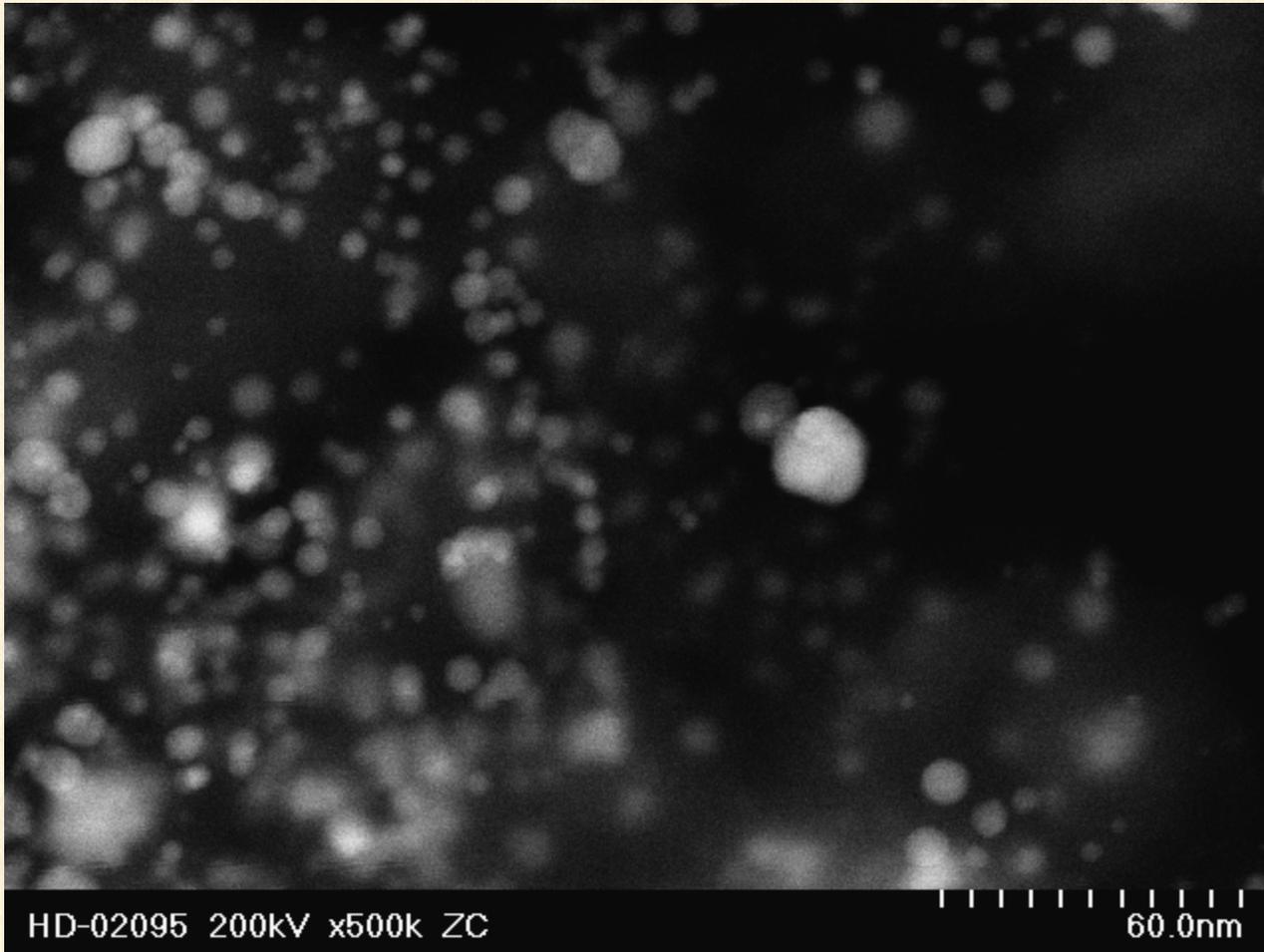
Established new ultramicrotomy facility for room-temperature sectioning



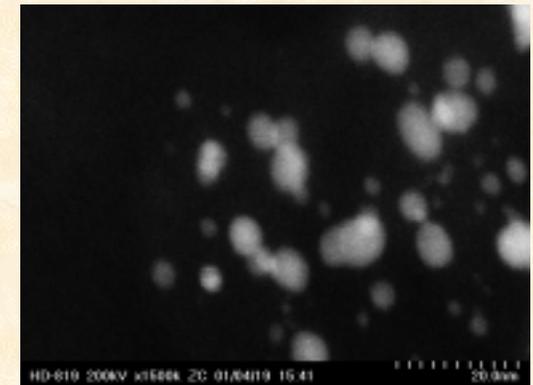
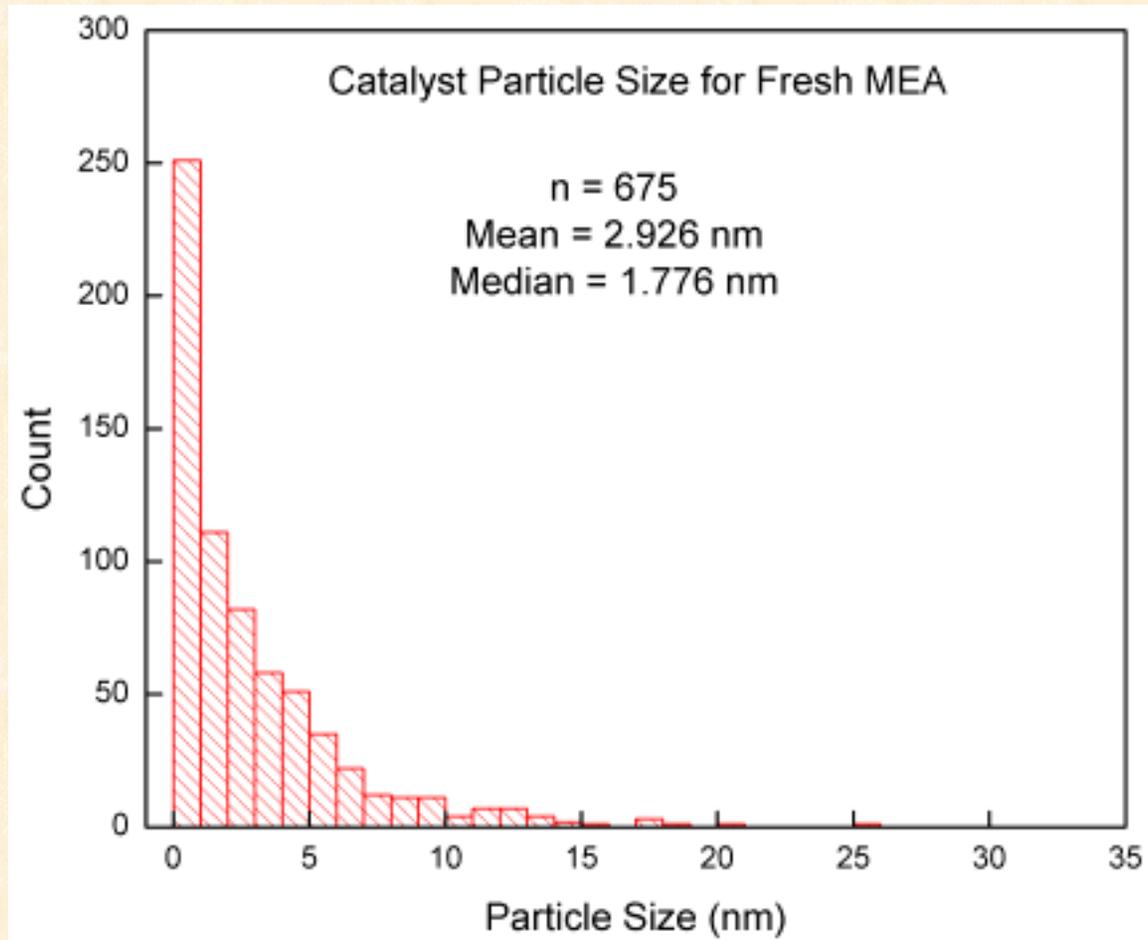
- Room temperature sections are 200 to 300 nm thick due to plastic properties of membranes – thinner would be better

Microstructural/microchemical characterization of MEA which failed after 1200 hours

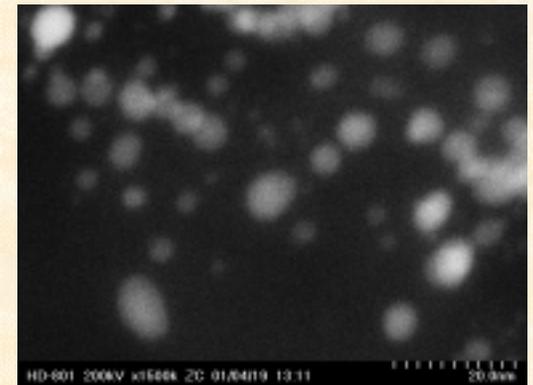
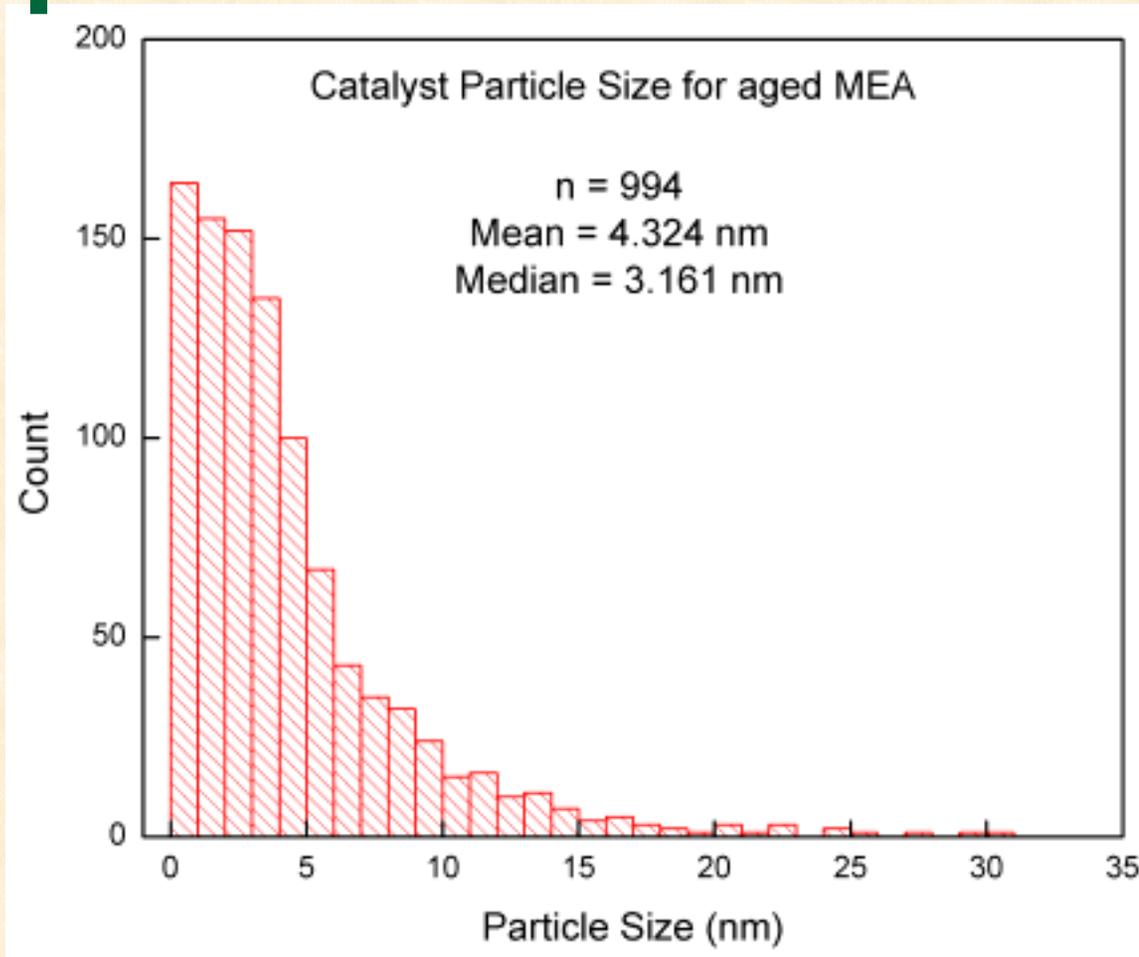




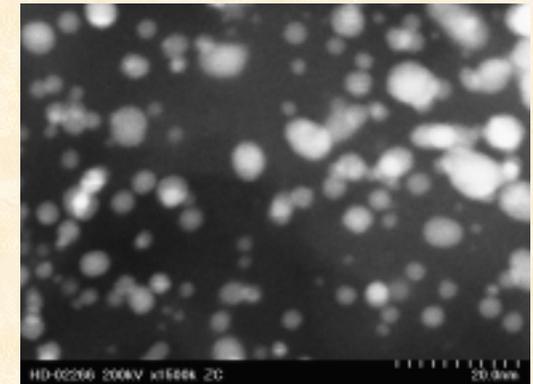
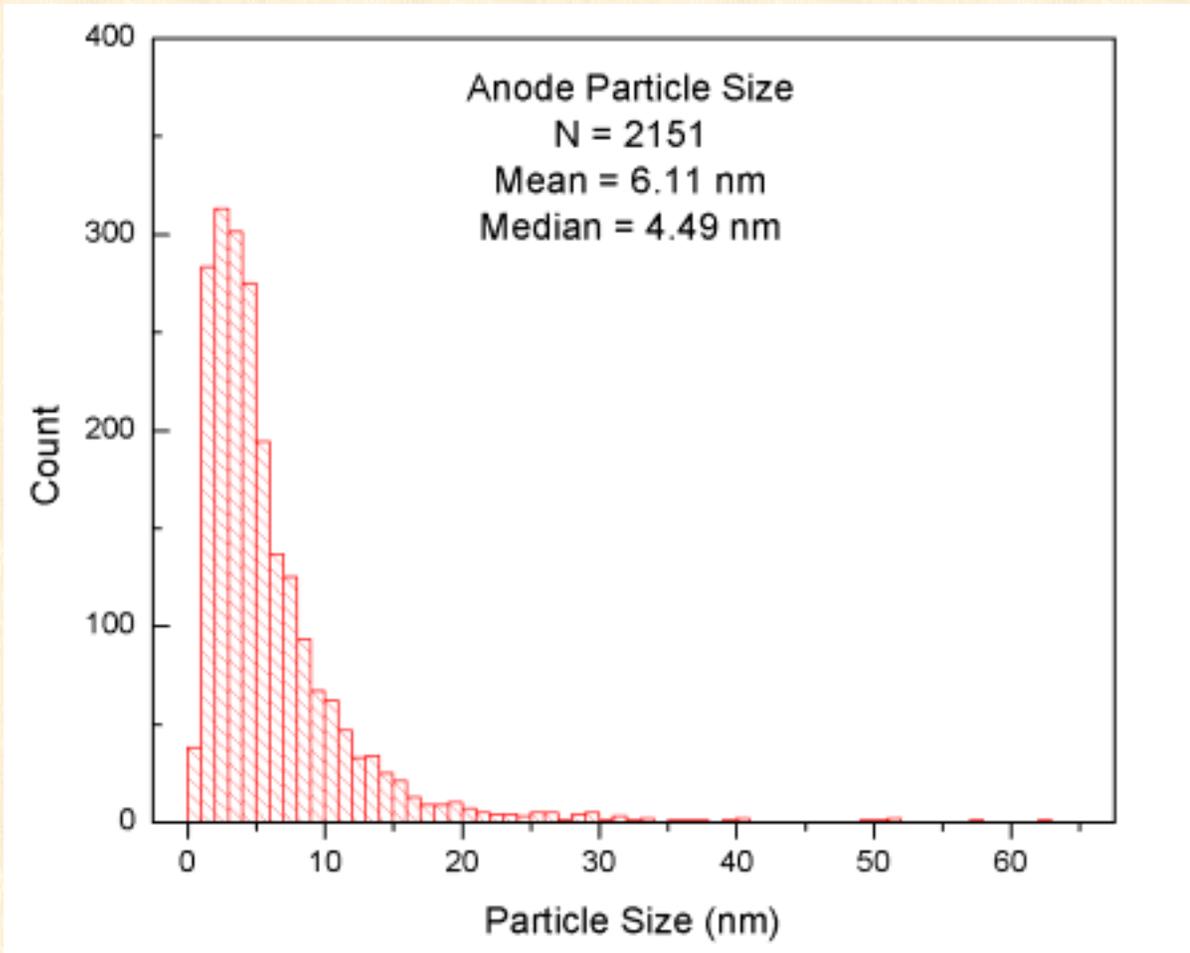
Catalyst Particle Size Distribution: Fresh MEA



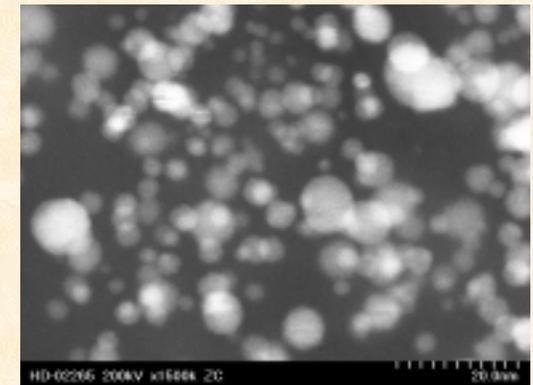
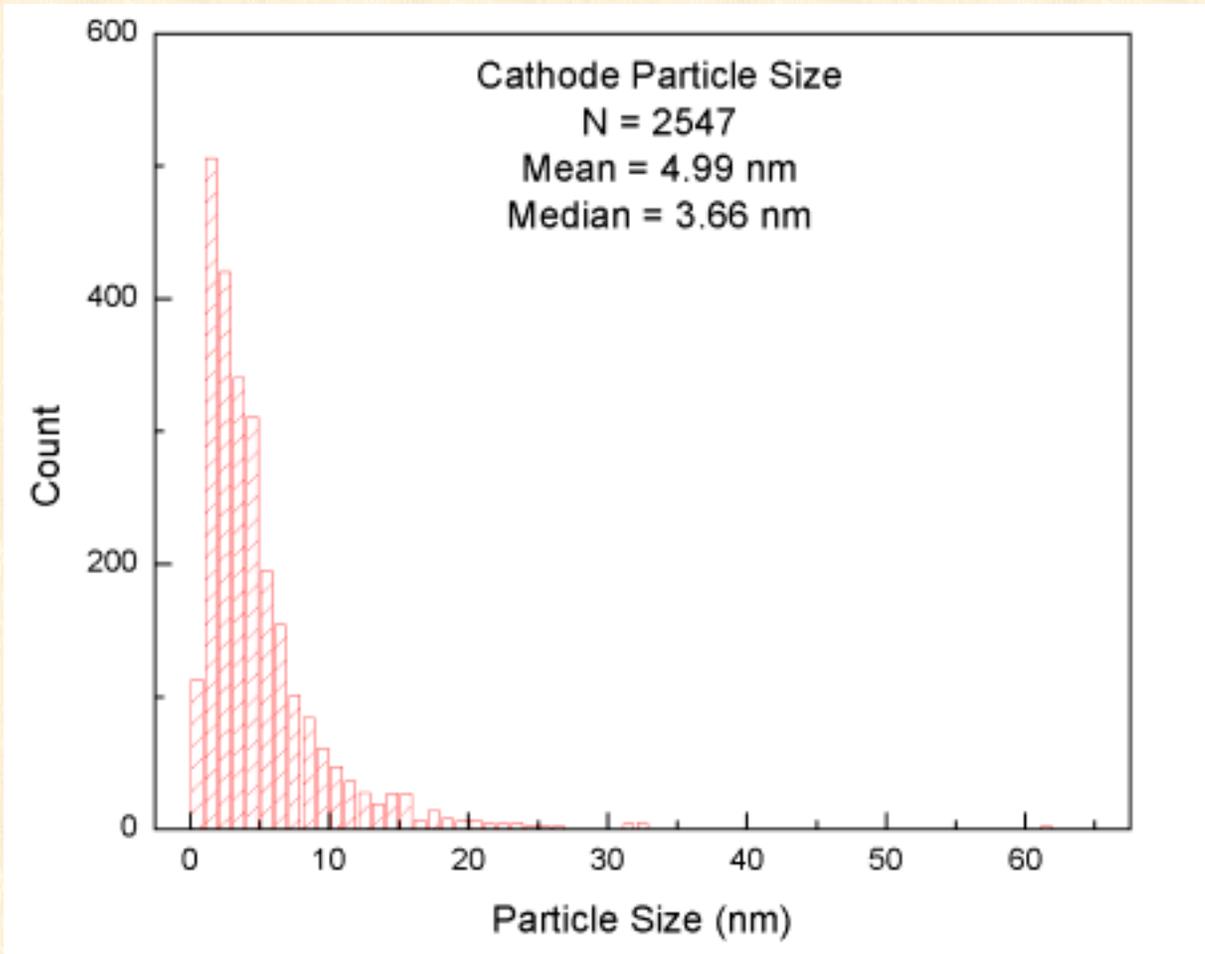
Aged 325 hours : minimal performance loss



Failed MEA: Anode



Failed MEA: Cathode



Catalyst Particle Sizing

	Mean (nm)	Median (nm)	Number of Particles (N)
Fresh MEA	2.93	1.78	675
After 325 Hours	4.32	3.16	994
1200 Hours Cathode	4.99	3.66	2547
1200 Hours Anode	6.11	4.49	2151

Collaborations

- **Third party MEA was tested to failure by W. L. Gore and Associates**
- **Began work with LANL on characterizing the microstructure of MEAs during the assembly process**

Plans and Future Milestones

- **Improve room temperature sectioning technique to allow for the routine preparation of thin MEA cross-sections**
- **Develop 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 2001 Review

- **“Effort is not focused enough on fuel cell issues”**
 - This effort is focused on baseline changes in catalyst morphology and PEM/electrode interfaces which lead to the degradation of performance of MEAs, one of the major issues for future successful operation of fuel cells in vehicles.

Response to Comments from 2001 Review

- **“Project lacks quantitative EDXA on cross sections”**
 - **Beam damage effects have limited the ability to collect reliable quantitative data.**
 - **Future cryo-microtomed samples may be more stable and allow reliable quantitative EDX data to be obtained.**