Quantitative Mesoscale Analysis of SOFC Electrodes Based on 3D Reconstructions using Xe-plasma Focused Ion Beam (PFIB) coupled with SEM

Research & Innovation Center

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Introduction

Electrode microstructures control performance of SOFCs and vary over the mesoscale

- Key factors limiting the commercialization of SOFCs:
  - I. High cost of manufacture
  - II. Poor reliability
  - III. Low durability
- A tradeoff exists between cost of manufacture and reliability / durability of electrodes:

Research-grade SOFC Samples	Commercial SOFC Samples
I. Higher-cost synthesis II. Ideal feedstock materials III. Produced in low quantities IV. Highly homogeneous microstructures	I. Lower-cost synthesis II. Less perfect feedstock materials III. Mass Production IV. Heterogeneous microstructures

- In commercial SOFCs, mesoscale (≈ hundreds of microns) microstructural variations are expected.
- How can we quantify the impact of mesoscale microstructural variations to processing / performance:

Measure Mesoscale Distributions of Microstructural Features → Quantify Variations in Local Electrochemical Activity → Feedback Loop to Improve Reliability / Durability at Low Cost

Objectives:

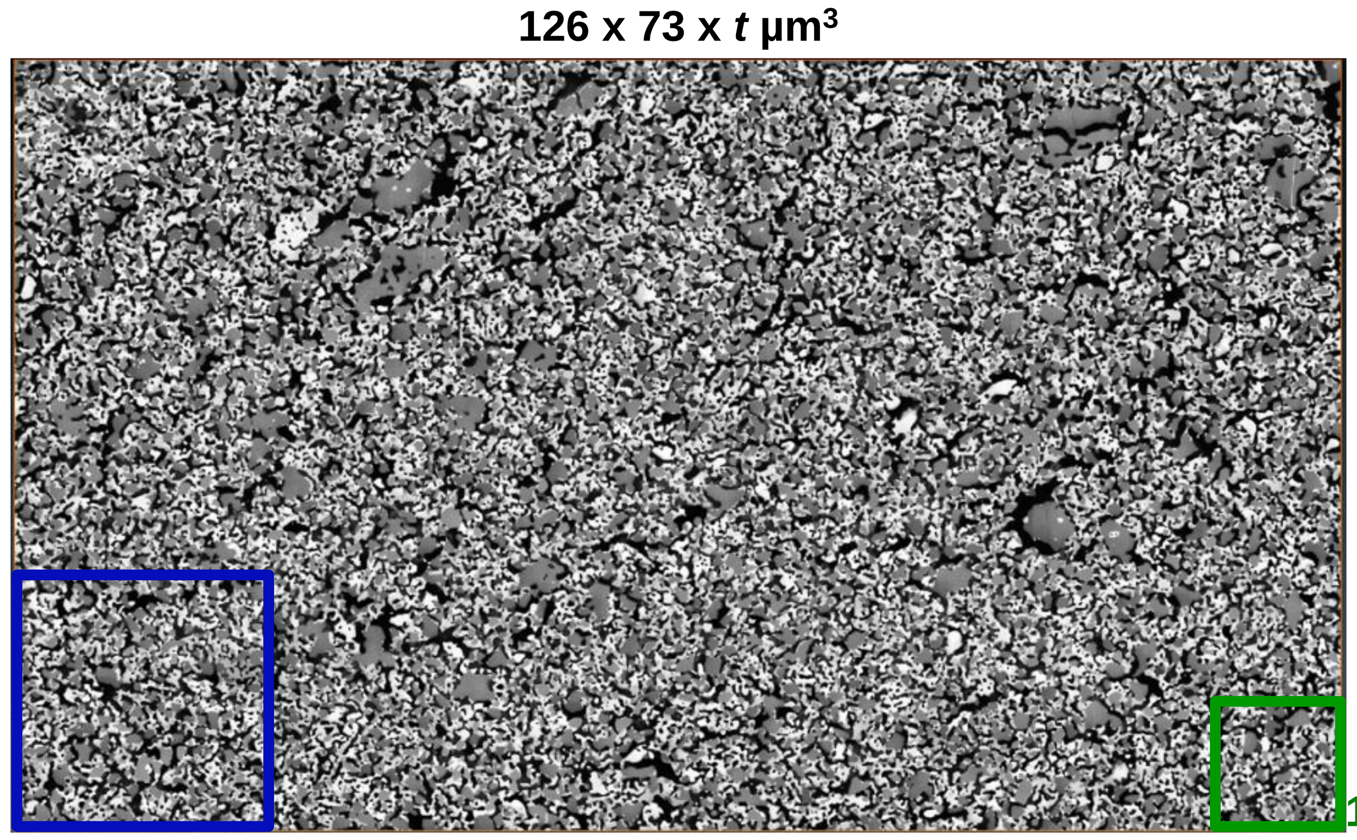
- To determine quantitatively the mesoscale distributions of microscale properties in SOFC electrodes using large volume / high resolution 3D reconstructions of commercial cells.
- To demonstrate correlations between outlier microstructural features and performance.

Why Xe-PFIB-SEM ?

PFIB can capture ≈ 50-100x Ga-FIB area and ≈10-50x nano-CT area in same amount of time

- High material removal rate using Xe-plasma Focused Ion Beam (Xe-PFIB or PFIB)
- Nano-scale resolution using Scanning Electron Microscopy (SEM)
- Unprecedented large volume / high resolution 3d reconstructions in standard collection times

Actual Xe-plasma-FIB Volume
126 x 73 x t μm³



t = thickness

Standard Nano-CT Volume 25 x 25 x t μm³

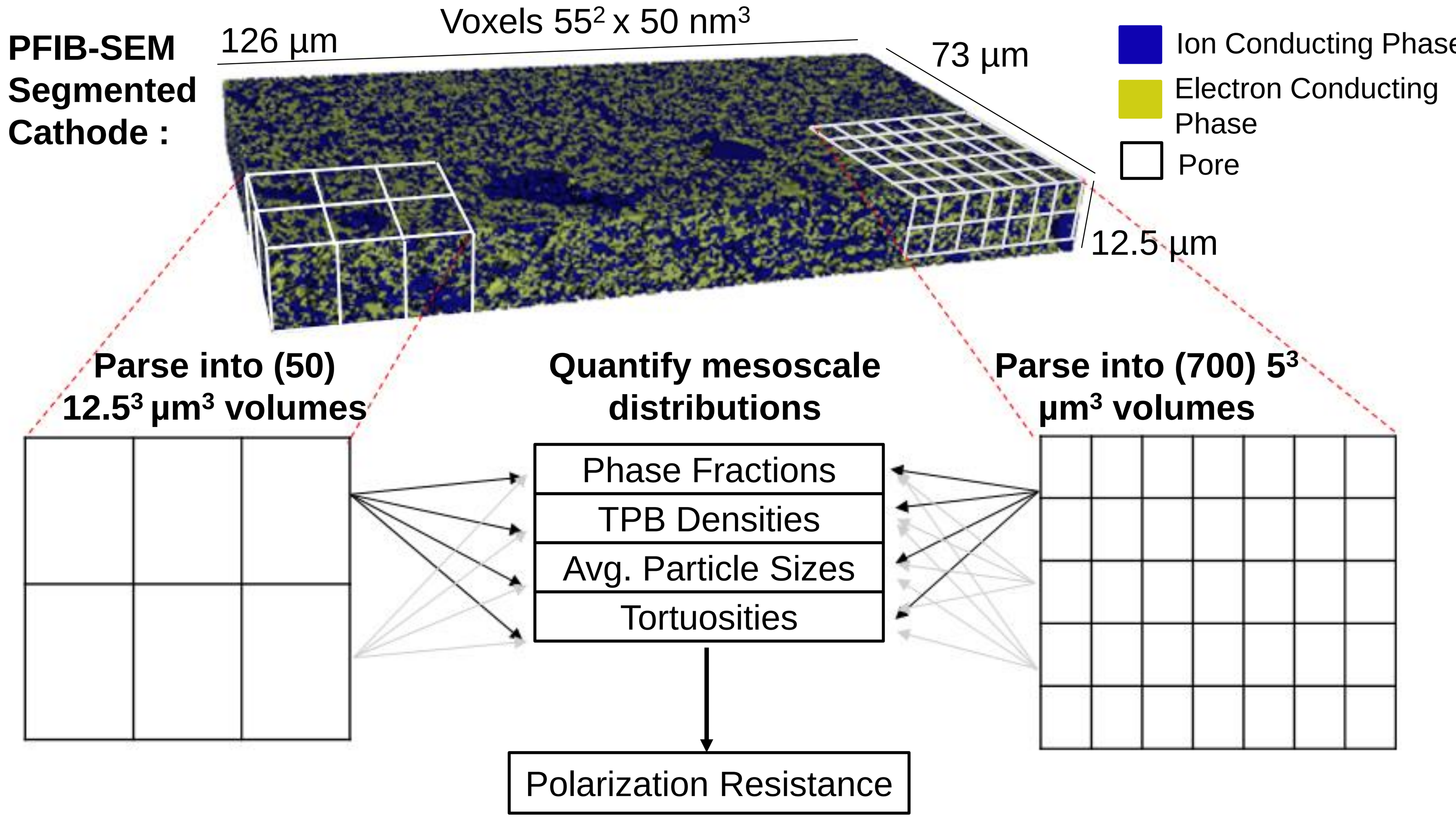
Standard Ga-FIB Volume 12.5 x 12.5 x t μm³

References:

[1] J. Joos et al., Electrochim. Acta, Vol. 82, pp. 268–276, 2012.
[2] W M Harris and W K S Chiu, J. Power Sources, Vol. 282, pp 552–561, 2015.
[3] J. Scott Cronin et.al., J. Electrochem. Soc., Vol.159, No.4, pp B385-B393, 2012.

Approach to Quantify Mesoscale Distribution

10s to 100s of microscale sub-volumes analyzed to generate mesoscale distributions of microstructural and performance parameters



Quantifying Microstructural Features:

- **≥ 5³ μm³** is a standard microscale volume that describes local electrochemical activity [1,2]
- 5 μm³ and 12.5 μm³ sub-volumes were analyzed using matlab codes for quantitative microstructural features

Quantifying Polarization Resistance:

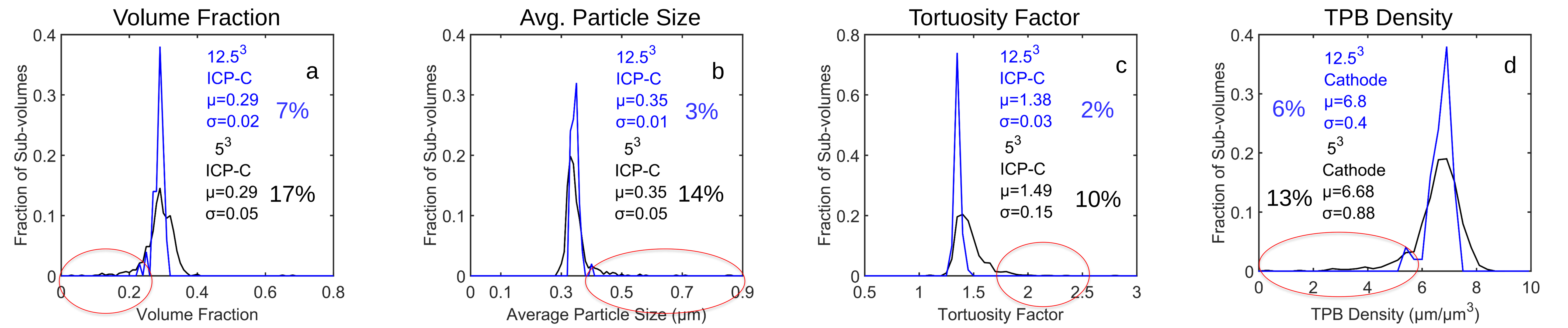
- Microstructural parameters taken as input to an effective medium theory model (TFV Model[3]) → outputs area specific polarization resistance (ASR)
- Distributions in local ASR are obtained and mapped

Assumption for TFV Model	➢ Cathode TPB-line Resistivity (800°C) ρ_{ct} = 1.9x10⁵ Ωcm ➢ ICP Conductivity σ^{O2-} = 0.055 Scm⁻¹
Microstructural Inputs for TFV Model	➢ Cathode Thickness ➢ Volume Fraction of Phases ➢ Average ICP Particle Size ➢ ICP Tortuosity Factor ➢ TPB Density

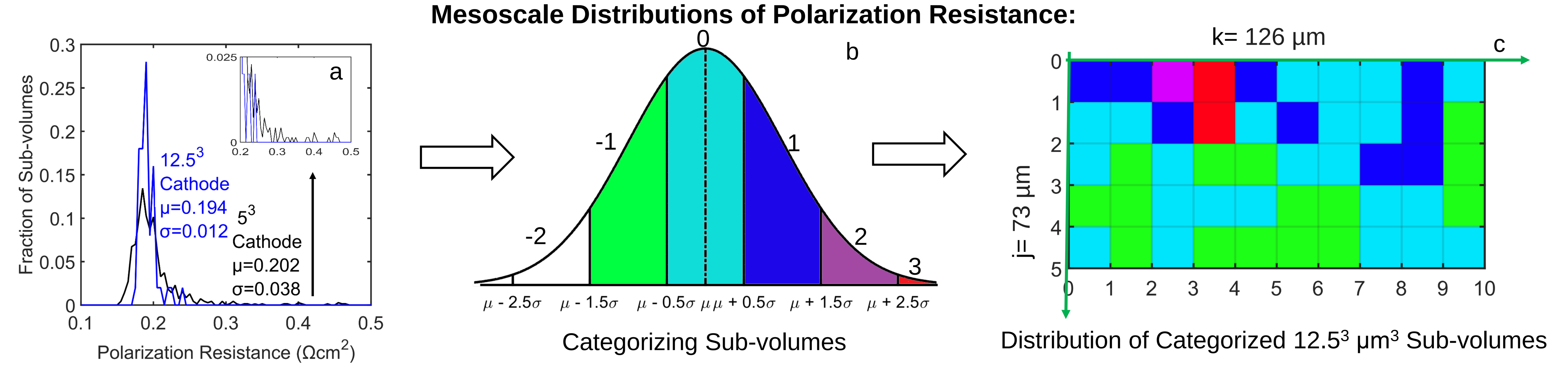
Results Visualization

Microstructural distributions are skewed, with one tail being more populated than the other

Mesoscale Distributions of Microstructural Parameters



Mesoscale Distributions of Polarization Resistance:



Local ASR values exist that are well outside the average values
Outliers that are expected to impact long term performance can be quantified using PFIB-SEM

Conclusions

- A commercial SOFC electrode was found to exhibit significant variations in its mesoscale distributions of local microstructural features.
- These variations in mesoscale microstructural features are found to result in significant variations in electrochemical activity throughout the electrode.
- The distribution of local polarization resistance around the mean indicates that some local areas are well outside the average.
- This work offers a path to analyzing the impact of local heterogeneities on long-term cell performance and to optimizing microstructural distributions.