Cell Degradation

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CELL DEGRADATION

OUTLINE

• Listing of several (most) of the cell degradation mechanisms
• General information and definitions of cell degradation mechanisms
• Examples of cell degradation for all fuel cell types
• Representative performance
• Focus on SOFC and PEMFC degradation
• Professor Mumm will present more detailed material in the next lecture
  • Thermal Stresses
  • Oxidation
General Degradation Mechanisms

- Corrosion
- Erosion
- Oxidation
- Reduction
- Mechanical failures
  - Thermal stresses
  - Chemical reaction and stresses
  - Propagation of initial defects

- Chemical Reaction
  - With other FC components
  - With reactant and/or product gases

- Sorption

- Chemical species poisoning
  - Carbon monoxide
  - Sulfur
General Degradation Mechanisms (cont’d)

- Dynamics – Cyclic operation
- Dehydration (PEMFC)
- Freezing (PEMFC, PAFC, AFC)
- Mass transport limitations due to:
  - Pore size reduction
  - Coarsening of porous materials
  - Contaminant plugging
- Carbon deposition (DMFC, SOFC, MCFC with HC in fuel)
- Morphology / structural changes
- Sintering (SOFC)
- Grain boundary growth
- Constituent element concentration within FC component

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Corrosion Definition:
- The chemical or electrochemical reaction between a material, usually a metal, and its environment that produces a deterioration of the material and its properties.

Erosion Definition:
- Destruction of metals or other materials by the abrasive action of moving fluids, usually accelerated by the presence of solid particles or matter in suspension.
- Destruction of materials by the abrasive action of the relative motion of adjacent cell components and materials.
- When corrosion and erosion occur simultaneously, the term erosion-corrosion is often used.
**Oxidation Definition:**
- Any chemical reaction that results in an increased atomic charge state (i.e., increase the valence by removing electrons)
  - \( 2 \text{H}^+ + \text{Cu}_2\text{O} \rightarrow 2 \text{Cu}^{2+} + 2 \text{e}^- + \text{H}_2\text{O} \)
- The chemical reaction that bonds oxygen to the molecular structure, which increases the charge state of adjacent atoms

**Reduction Definition:**
- Any chemical reaction that results in decreased atomic charge state (i.e., decrease the valence by adding electrons)
  - \( 4 \text{H}^+ + \text{NO}_3^- + 3\text{e}^- \rightarrow \text{NO} + 2 \text{H}_2\text{O} \)
- The chemical reaction that removes bonded oxygen from the molecular structure decreasing the charge state of adjacent atoms

**Oxidation-Reduction (Redox):** a chemical reaction between two substances in which one substance is oxidized and the other is reduced
Mechanical stress (and strain)

- The physical pressure, pull, or other force exerted on one thing by another
- The action on a body of any system of balanced forces whereby strain or deformation results
  - Thermal gradients can lead to and/or initiate these mechanical stresses
  - Chemical reaction changes volume and can lead or contribute to mechanical stresses
  - Diffusion of species changes mechanical properties (and volume) and can lead or contribute to mechanical stresses
  - Propagation of initial defects (pin-holes, cracks)
- Failure can be catastrophic (e.g., crack) or gradual (e.g., creep)
Chemical Reaction

- The reciprocal action of chemical agents upon each other leading to chemical change
- In fuel cells chemical reactions can occur between the various FC components
  - Anode
  - Cathode
  - Electrolyte
  - Interconnect
  - Bipolar plates
- Chemical reactions can occur between fuel cell components and reactant and/or product gases (H₂, O₂, H₂O)
- Original functionality of the fuel cell component(s) is either lost or impaired

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Sorption

• To gather on a surface either by absorption, adsorption, or a combination of the two

• **Absorption**: to suck up and assimilate by chemical or molecular action

• **Adsorption**: to hold a gas, liquid or dissolved substance on a surface in a condensed layer (primarily physical action)

• In a fuel cell, compounds (e.g., carbon monoxide, sulfur) can be “preferentially” absorbed and/or adsorbed and subsequently very challenging to remove
  • Temperature driver (energy barrier)
  • Concentration gradients

• This sorption prohibits or inhibits the original functionality of the sites affected by sorption

• Chemical species poisoning by CO (for platinum catalyst of PEMFC and PAFC) and sulfur (all FC types) is a subset
Dynamics – Cyclic operation

- Typical thermal gradients, concentration gradients are large during cyclic, dynamic operation
  - Versus time
  - Versus space
- Stresses (physical, chemical, electrochemical) are exaggerated under cycling conditions
- Small non-uniformities, defects can readily propagate under dynamic cycling operation
- Dissimilar thermal expansion coefficients lead to stresses that are accentuated during cyclic operation
Dehydration (PEMFC)

- Performance of a PEMFC depends upon presence of water in membrane
- Dehydration can occur since water is dragged through membrane (osmotic drag) during typical operation
- Operating conditions of PEMFC have impact on dehydration
  - High power density operation
  - Lack of sufficient reactant humidification
- MEA (membrane electrode assembly) structure has impact
  - Back-diffusion of water affected by porosity, intimacy of contacting, hydrophobic/hydrophilic nature of MEA
- Design of system has a large impact
  - Humidifier operation
  - Dependence on back diffusion
  - Directions of reactant/product flows in stack
  - Channels for water bypass
Freezing (AFC, MCFC, PAFC, PEMFC)

- Phase change from liquid to solid by loss of heat
- Electrolyte itself changes state in fuel cells containing a liquid electrolyte (AFC, MCFC, PAFC)
  - Mechanical stress
  - Repetitive phase change can affect functionality
- Absorbed water constituent can change phase (PEMFC membrane absorbs much water)
- Occurs to all fuel cell product gases (H₂O) when subjected to temperatures or operating at temperatures below the freezing point of water
  - Challenging for this to occur with high temperature fuel cells when operating
  - Affects all fuel cells during maintenance, full thermal cycling in cold climates
Mass transport limitations due to:

- Pore size reduction due to thermal or mechanical creep
- Coarsening of porous materials – usually thermally induced changes in morphology
- Contaminant plugging (e.g., dust)

Carbon deposition (DMFC, SOFC, MCFC with HC in fuel)

- Formation of carbon (versus carbon monoxide or carbon dioxide gas) can readily occur (equilibrium driver for formation) in some fuel cells
  - Temperature
  - Pressure
  - Presence of catalyst
  - Reactant gas composition
- Carbon can occupy active sites and plug pores
Morphology / structural changes

- Porosity of electrodes can be lost during normal operation of a fuel cell
- Intimacy of contacting between fuel cell components can be lost due to structure changes
- Amount of triple phase boundary (TPB) can be lost due to changes in morphology

Sintering (SOFC)

- Growth of crystalline structure, removal of grain boundaries and other processes associated with sintering can occur at very high temperatures in fuel cells
- This impacts functionality of fuel cell components and can be undesirable
Grain boundary growth

• Within the crystalline structure of a fuel cell component, grain boundaries exist that can grow in length and width during normal operation.

• Often the grain boundaries do not have the same functionality as the bulk material – leads to performance degradation.

Constituent element concentration

• Within the structure of a fuel cell component (e.g., electrode, electrolyte) elements that comprise the overall molecular composition may become spatially concentrated in certain locations leading to loss of functionality.
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Morphology change example (creep)

- Porous Ni anode – works well for MCFC, SOFC electrochemical operation
- But porous nickel creeps significantly under compressive load during operation
  - Mechanical stresses lead to cracking of matrix/electrolyte materials
  - Decreased anode performance due to a reduction of active area
- Several means have been attempted to reduce creep
  - Use of Ni coated ceramic powders
  - Impregnation of LiAlO$_2$ with Ni
  - Ni sintered from Ni-Cr and Ni-Al
- Only Ni sintered from Ni-Cr or Ni-Al appear to meet current requirements
- Primary application is with MCFC anodes
Corrosion example:

- NiO (made by oxidation of porous Ni) is used as a cathode material in MCFCs
- Corrosion is due to the low solubility of NiO (<30 ppm) in carbonate melts
- Dissolved Ni ions precipitate as Ni metal when they encounter more reducing atmospheres near the anode
- The concentration gradient thus produced drives NiO transport
- Net result of continuous cathode dissolution and Ni precipitation in the matrix.
  - Cathode material loss
  - Internal cell shorting
- Alternative cathode materials under investigation (doped LiFeO$_2$, Li$_2$MnO$_3$, and LiCoO$_2$) as a substitute for NiO
  - Electrical conductivities are low, but, significantly improved by doping
  - Still lower performance compared to NiO
Cell Degradation

**Mechanical stress example:**

- Many fuel cells are sealed around the edges by compressive seals
- Compression itself produces mechanical stress
- Different mechanical characteristics of the seal areas generate mechanical stress
- Differential expansion characteristics of the plate and seal materials force edges of the cell to experience mechanical stress
  - Changes in electrolyte volume
    - PEMFC water mass fraction dependence
    - MCFC freezing, melting
    - PEMFC freezing
  - Thermal expansion
    - Primarily a problem with high temperature fuel cells (e.g., MCFC, SOFC)
- Cell edges
Thermal stress examples:

- Thermal stress is generated by non-uniform temperature distribution (temperature gradients)
  - Cell level
  - Stack level
  - System level
- Thermal cycling (dynamics)
  - Primarily an issue with high temperature fuel cells (SOFC, MCFC)
- Differential thermal expansion amongst cell components
  - Primarily an issue with high temperature fuel cells (SOFC, MCFC)
  - Large temperature change often required
- Thermal gradients and differential expansion can each lead to mechanical cracking of cell components
  - Electrolyte and matrix are a particular concern
  - Can result in gas leakage, lower efficiency, and a reduction in stack life
Corrosion examples:

- Corrosion impacts mechanical properties, accelerates electrolyte loss and increases ohmic resistance
- Corrosion is a serious problem for the bipolar plate
- Reducing (anode) environment produces a thick bi-layered oxide scale on unprotected austenitic stainless-steels
  - Inner layer is rich in iron and chromium. Ni rich metallic islands are also present in the inner oxide scale
  - Outer scale consists primarily of lithiated, large crystalline iron oxide
- Oxidizing (cathode) environment is less problematic with corrosion of 316S producing a thinner multi-layered structure ($\text{LiFeO}_2$ outer layer, Cr-rich inner layer)
- Surface protection (e.g., Ni coating) is required to prevent corrosion
  - Ni can be applied by either plating or cladding
**Corrosion**

- In a battery or fuel cell, electrochemistry happens where you want it
- Corrosion is electrochemistry happening where you don’t want it
  - All M/MOx couples at $E^\circ < 0.4V$ are corroded even in caustic solutions:
    - $O_2 + 2 H_2O + 4 e^- \rightarrow 4 OH^- \quad E^\circ = 0.40$ (caustic environment)
    - $O_2 + 4 H^+ + 4 e^- \rightarrow 2 H_2O \quad E^\circ = 1.23$ (acidic environment)
      - So acid does even better

- Electrochemical driving forces in a fuel cell can corrode fuel cell materials
  - changing metals to metal oxides on the one hand and metal oxides to metals on the other causing them to lose original functionality
Metal Corrosion

- Metal oxides are lower density (higher volume) than their corresponding metals
  - So oxide formation opens “blossoms” of corrosion that increase spreading of corrosion
- Density change leads to mechanical stresses as well
- Salt spray is worst since the salt itself is electrolytic
- Some oxides (e.g., Cr$_2$O$_3$) form impervious oxide coats, slowing further O$_2$ attack
Metal Corrosion

• Note on Sacrificial Anodes
• Structural metals like Fe can be protected by more active (lower $E^\circ$) metals like Mg
  • If conductive contact is made, $O_2$ gets reduced (to $H_2O$) on Fe by $e^-$ released from Mg instead of the Fe
• Replacing the active metal plate is cheaper than an entire rusted ship

\[ Mg \rightarrow Mg^{2+} + 2e^- \]
\[ O_2 + 2H_2O + 4e^- \rightarrow 4 OH^- \]
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Representative Performance Degradation and Lifetime

- **AFC**
  - State-of-the-Art: 20-25mV reduction per 1,000 hours of operation
    (-25mV/1000hr)
  - 4,000 – 8,000 hour lifetime

- **MCFC**
  - Historically: ~20mV reduction per 1,000 hours of operation
    (-20mV/1000hr)
  - State-of-the-Art: ~5mV reduction per 1,000 hours of operation
    (-5mV/1000hr)
  - 15,000 – 25,000 hour lifetime
  - Expected improvements to –2mV/1000hr (40,000 hr lifetime)

- **PAFC**
  - 2-3mV reduction per 1,000 hours of operation (-2mV/1000hr)
  - 40,000 – 50,000 hour lifetime

- **NOTE:** One-Year, continuous operation = 8,760 hrs.
Representative Performance Degradation and Lifetime (cont’d)

• **PEMFC**
  - State-of-the-Art: ~20mV reduction per 1,000 hours of operation (-20mV/1000hr)
  - 4,000 – 8,000 hour lifetime

• **Tubular SOFC**
  - Historically: ~3.5mV reduction per 1,000 hours of operation (-3.5mV/1000hr) (cells operated >100,000hrs, stacks >70,000 hrs)
  - State-of-the-Art: ~1-1.5mV reduction per 1,000 hours of operation (-1.5mV/1000hr) (recent shorter term tests)
  - 50,000 – 100,000 hour lifetimes possible

• **Planar SOFC**
  - Early evidence (not much data available): -25mV/1000hr
  - 4,000 – 8,000 hour lifetime
  - Many improvements expected in near future
How is degradation observed and handled?

- Voltage drop is measured/observed for constant current demanded (supplied by the fuel cell)
- Typically lower current (or power) demanded of the cell in response to lower observed voltage
  - Step increase in voltage occurs
  - Leads to increase in thermal output
- MCFC example presented below
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SOFC cell component general requirements

- Electrolytes and interconnects must be stable in both oxidizing and reducing environments
  - Chemical stability
  - Morphological stability
  - Dimensional stability

- All cell components:
  - No significant changes in volume between room temperature and the fabrication temperature.
  - Chemically stable to limit chemical interactions with other cell components.
  - Similar thermal expansion to minimize thermal stresses
    - Cracking
    - Delamination
  - High strength and durability
  - Easy to fabricate
  - Relatively inexpensive
  - Characteristics hold during thermal cycling and fabrication
SOFC ELECTROLYTE

- Stability in oxidizing and reducing environments
  - YSZ shows very good redox resistance
  - Other electrolytes have some challenges
- **Example: Sm$^{3+}$ or Gd$^{3+}$ doped cerium oxide (CeO$_2$)**
  - Electronic conductivity under reducing conditions
  - Reduction of Ce$^{4+}$ to Ce$^{3+}$ with low oxygen partial pressures
CATHODE DEGRADATION

- Lack of thermal stability
- Lack of resistance to oxidation (at temperatures up to 1000°C a very big challenge)
- Thermal expansion not comparable to electrolyte (and/or interconnect)
- Non-reactive with interconnect materials (especially a problem with metal interconnects)
- Loss of porous microstructure for gas diffusion
- Current choice: lanthanum manganite (LaMnO$_3$) doped with an appropriate amount of Ca or Sr
  - Not a good conductor of either electrons or ions – just sufficient
  - Reasonable material for stable operation, resistance to degradation mechanisms above
  - Real need/opportunity for advances in design of materials for cathode
CATHODE DEGRADATION (cont’d)

• At high temperatures, lanthanum manganite and YSZ can react to form insulating products (e.g., La$_2$Zr$_2$O$_7$, SrZrO$_3$, CaZrO$_3$)

• Depends on sintering temperature and time, La to Sr (Ca) ratio, and dopants

• A pure acceptor substituted lanthanum manganite electrode behaves like a metallic electrode
  • Reaction takes place only at the triple phase boundary (TPB)

• Mixed conductors are being considered and tested as cathodes to widen the TPB

• When mixed conductors are used the reaction takes place on the TPB and on the whole surface of the cathode

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ANODE DEGRADATION
• Lack of stability in reducing environments is one degradation mechanism
• Nickel-YSZ cermet - almost exclusively used
  • YSZ incorporated into the anode as the supporting framework for the Ni
  • Thermal expansion coefficient lowered
  • Need to prevent Ni coarsening
• Ni coarsening
  • Reduction in the TPB
  • Reduced electrical conductivity
• Delamination of anode from electrolyte
• Oxidation of anode removes functionality – recoverable (?)
• Lack of “redox” resistance
  • Oxidation often occurs during start-up, shutdown procedures when hot anode can be exposed to oxidant (e.g., air)
  • How well does the anode recover from oxidation?
ANODE (cont’d)

- Ni can be sintered in the anode due to high interfacial energy between Ni and YSZ
- Typically there is limited common interface between the Ni and YSZ (essentially Ni wetting on YSZ)
- Modification of YSZ with lower surface energy oxides, such as MgO, TiO$_2$, AND Cr$_2$O$_3$, can reduce Ni coarsening
- Anodic overpotential has been decreased by placing a mixed ionic-electronic conductor such as (CeO$_2$)$_{0.8}$(Sm$_2$O$_3$)$_{0.2}$ between the anode and electrolyte
- Other ideas for better SOFC anode performance:
  - Replacing YSZ with ceria
  - Incorporate precious metal catalysts
  - Improve distribution of YSZ and Ni in the anode
INTERCONNECTS

- Electrically connect anode to cathode of adjacent cells in series and physically separate of anode and cathode chambers (reducing and oxidizing environments)
  - High density (>94%Th.D.)
  - High electrical conductivity
  - Low ionic conductivity
  - Good thermal, mechanical, chemical stability
  - Match thermal expansion (electrodes and electrolyte)

- Very difficult criteria
- Currently use ceramic interconnects
- Metal interconnects under development and initial use
INTERCONNECTS (cont’d)

- Acceptor (Mg$^{2+}$, Sr$^{2+}$, Ca$^{2+}$) substituted lanthanum chromite is main candidate material

- But, LaCrO$_3$ difficult to sinter to high density in air, poor mechanical strength, unstable under severely reducing environments

- Several techniques for sintering high density LaCrO$_3$
  - Add sintering aid (e.g., fluorides)
  - Highly reactive powders
  - Firing LaCrO$_3$ between Cr$_2$O$_3$ plates
  - Add alkaline earth elements (e.g., Ca, Sr or Mg), or transition metals (e.g., Zn or Cu), or low melting point eutectic compositions
INTERCONNECTS (cont’d)

- Recent development: transient liquid phase sintering in slightly non-stoichiometric Ca-doped LaCrO$_3$ (LCC)
- LCC, however, has poor dimensional stability under reducing atmospheres
- Perhaps use Sr-doped LaCrO$_3$, but, very difficult to sinter in air
- Active area of research since lanthanum chromite is dominant, but, has several problems/issues
  - Instability in reducing environments
  - Expansion upon reduction
  - Mechanical strength reductions
  - Phase segregation in microstructure
  - Sintering difficulty
  - Cost
INTERCONNECTS (cont’d)

- **Metal interconnects**
  - Lower cost
  - Ease of manufacture/forming
- **Main problem is metal oxidation (degrades conductivity)**
  - Add protective oxidized layer - slow growing
- **Matching thermal expansion is difficult – mechanical stress degradation**
- **Oxidation is a primary degradation mechanism**
  - Oxide growth rate
  - Conductivity of oxide
- **Main candidates**
  - Ni/NiO - Conductivity good, CTE too high
  - \( \text{Cr}_2\text{O}_3 \)-forming alloys - relatively low oxide growth rate and relatively high electrical conductivity
  - Cr-based alloys such as the plansee alloy Cr-5Fe-1Y\(_2\)O\(_3\) wt.% - designed to match the thermal expansion of zirconia-based electrolyte, cheaper, but limited to lower temperatures for longevity
PEMFC DEGRADATION

PEMFC MATERIALS DEGRADATION - Membrane

• Mechanical strength of the membrane
  • Reinforced vs. non-reinforced
  • Membrane thickness

• Membrane failure characteristics are exhibited by an increase of $H_2$ crossover rate

• Non-reinforced membranes often exhibit immediate sudden failure

• Reinforced membranes often show gradual increases in gas crossover until pinhole failures occur

• Fluoride is typically released and present in the product water. Measurements do not correlate well with failure and total percentage lost is low, indicating that failure is highly localized

Liu, Ruth, and Rusch, GORE, 2001
PEMFC Membrane – Freezing example

- Bulk of the polymer is fluorinated - highly hydrophobic character

- But, the sulfonic acid sites are hydrophylllic
  - water content is proportional to the ionic sites (desire to maximize)
  - significant properties of the membrane (conductivity, gas permeability, and mechanical properties) are dictated by water content

- Membrane typically experiences between 20% and 50% weight gain by absorbing moisture

- Freezing of a wet membrane may damage polymer structure
  - Significant recent observations lead one to believe freezing is quite harmful to current PEM membranes

- Freezing can lead to significant additional mechanical stresses throughout the fuel cell stack and system
  - Coolant system freezing a major concern
PEMFC Membrane – Mechanical Stress Example

- Significant properties of the membrane are dictated by water content
  - Ionic conductivity
  - Volume
  - Mechanical strength
  - Thermal expansion coefficient

- Dynamic cycling of water (or gas phase humidity) content can lead to mechanical stresses
  - Volume changes
  - Thermal expansion changes

- Repetitive humidification / drying cycles may lead to lower steady state water content
  - Steady-state volume is less than original design leading to mechanical stress
PEMFC Membrane – Drying Example

• Ionic conductivity of the membrane is dictated by water content
• Without water present in membrane fuel cell ceases to conduct ions
• Can lead to temperature gradients due to lack of conductivity (electrochemistry) in some places and accelerated electrochemistry in others
  • Could increase the drying action
  • Could eventually destroy cell
PEMFC DEGRADATION

PEMFC Backing Layer degradation examples

• **Very porous layer that sandwiches polymer membrane**
  - act as a gas diffuser
  - provide mechanical support
  - provide an electrical pathway for electrons

• **Typically carbon-based**
  - Cloth, non-woven pressed carbon fiber or felt-like material

• **Incorporates a hydrophobic material, e.g., Teflon®**
  - prevent water from “pooling” within pore volume
  - gases move freely to the catalyst sites
  - facilitates product water removal on the cathode as it creates a non-wetting surface within the passages of the backing material

**Degradation by:**

• Compressive stress, pressure
• Oxidation at surface / interface
PEMFC DEGRADATION

PEMFC Electrode – Catalyst Layer
- Intimate contact with the membrane and the backing layer
- Catalyst integrated with a binder forms the electrode
  - electrode structure either applied to the membrane or to the backing layer
- Intimacy of the catalyst particles and the membrane is critical for optimal proton mobility
- Binder functions
  - fixes the catalyst particles within a layered structure
  - contribute to the overall architecture of the electrode
- Architecture / Morphology has a direct bearing on performance

Degradation Mechanism(s):
- Delamination (loss of intimate contacting)
- Poisoning (preferential occupation, chemisorption of CO, sulfur at catalyst electrochemical sites)
- Morphology modification – due to mechanical or thermal stress
- Oxidation
- Corrosion

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PEMFC Developments – Impacts on Degradation:

- Typical proton conduction process drags water through membrane
- Ionic conductivity and other properties of membrane depend upon water content
  - Need for humidified reactants
- Recent high temperature PEMFC development
  - Operation in the 160°C range
  - Use new ion exchange membrane, polybenzimidizole (PBI)

Degradation Mechanism Impacts:

- **Poisoning:** Higher operating temperature eliminates CO poisoning by eliminating CO occlusion of the platinum sites
- **Drying:** PBI requires significantly lower water content to facilitate proton transport
  - easier water management
- **Freezing:** May be less susceptible to freezing damage
- **Dynamic cycling:** Certainly less susceptible to drying damage and less dynamic cycling of water content may lead to reduced mechanical stress
- **Mechanical and Thermal Stress:** May increase due to larger temp. range
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