CORROSION-RESISTANT NON-CARBON ELECTROCATALYST SUPPORTS FOR PEFCS

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Date: 6/7/2016

Project ID # FC145

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Overview

Timeline and budget

Competitively selected project

- Project start date: 03/01/16*
- Project end date: 02/28/19
- Total project budget: $ 3,397,431
  - Total recipient share: $ 397,431
  - Total federal share: $ 3,000,000
  - Total DOE funds spent**: < $ 50,000

Partners

- Project lead: IIT, Chicago
- Partners (sub-contractors):
  – Nissan Technical Center, North America
  – University of New Mexico

* Official date of contract from DOE. Issue of sub-contracts were finalized on April 15th 2016. Kick-off meeting held on April 21st 2016

** As of 3/31/16

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Barriers and DOE target

- Barriers to be addressed:
  - Durability
  - Performance
- Technical targets:

	Units	2020 Target
Loss in catalytic (mass) activity[a,b]	% loss	<40
Loss in performance at 0.8 A/cm^2 [a]	mV	30
Loss in performance at 1.5 A/cm^2 [b]	mV	30
Mass activity @ 900 $mV_{iR\text{-}free}$ [c]	A/mg_{PGM}	0.44

[a]-Table E1, [b]-Table E2; Appendix E of FOA; [c] DOE protocol per appendix E of FOA

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Impact of carbon corrosion on PEFCs

Carbon is mainly used as an electrocatalyst support due to its:

- High electrical conductivity (> 20 S/cm)
- High BET surface area : 200 - 300 m^2/g
- Low cost

Electrochemical oxidation of carbon occurs during fuel cell operation

- $C + 2H_2O \rightarrow CO_2 + 4H^+ + 4e^-$ E^o = 0.207 v vs. SHE

Carbon corrosion is accelerated:

- During start/stop operation (cathode carbon corrosion)
- Under fuel starvation conditions (anode carbon corrosion)

Kinetic and ohmic losses result due to:

- Pt sintering and loss of contact between Pt and C

Mass transport losses occur due to

- Formation of hydrophilic groups => flooding

Relevance

Research objectives

- Conducting, doped, non-PGM metal oxides (electron conductivity >0.2 S/cm)
- High surface area(>70 m^2/g)
- Exhibits SMSI with Pt
- Corrosion resistant (DOE 2020 targets)
- High electrocatalyst performance (DOE 2020 targets)

Metal oxide	Stable potential window (vs. SHE) (pH 0-1)	Manifestation of SMSI	Possible dopants
TiO_2 (4+, 60.5 pm)	-0.4 - 2.2 V	Yes	Nb (5+, 64 pm), Ta (5+, 64 pm), Mo (6+, 59 pm), W (6+, 60 pm)
Nb_2O_5 (5+, 64 pm)	-0.2 - 2.2 V	Yes	Mo (6+, 59 pm), W (6+, 60 pm), Tc (7+, 56 pm), Re (7+, 53 pm)
Ta_2O_5 (5+, 64 pm)	-0.7 - 2.2 V	Yes	Mo (6+, 59 pm), W (6+, 60 pm), Tc (7+, 56 pm), Re (7+, 53 pm)

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Research objectives: Technical targets

Metric	Units	SoA (Pt/C)*	SoA (Pt/RTO)	Proposed approach status (Pt/TiO_2-Ta)**	End target	DOE 2020 target
Total PGM content	$g\ kW^{-1}$	0.55	0.55	Not Available	0.25	<0.125
Total PGM loading	$mg\ cm^{-2}$	0.4	0.4	0.6	0.25	<0.125
Voltage at 1.5 $A\ cm^{-2}$ (air)	mV	0.45	0.48	0.3	0.55	N/A
Loss in mass activity [a,b]	% loss	32	33	<10%	<5%	<40
Voltage loss at 0.8 $A\ cm^{-2}$ [a]	mV	81	9	< 15	<10	30
Voltage loss at 1.5 $A\ cm^{-2}$ [b]	mV	182[+]	20	N/A; 20 mV at 1Acm^{-2}	<20	30
Mass activity@900 $mV_{iR\text{-}free}$ [c]	$A\ mg^{-1}_{PGM}$	0.07	0.07	ca. 0.05	0.3	0.44

[a]-Table E1, [b]-Table E2; Appendix E of FOA; [c] DOE protocol per appendix E of FOA; **Pt/C refers to Pt/Graphitized Ketjen Black tested at NTCNA; **Results from entirely un-optimized MEAs run primarily to test stability. [+]Pt/HSAC durability is much worse – MEA does not run beyond 0.5 $A\ cm^{-2}$ after start-stop cycling.*

Data from MEA in a PEFC

Relevance

Research objectives: 1^{st} year milestones

Q1
- 2g Ta-doped TiO_2
- B.E.T. surface area >30 m^2g^{-1} ; Electronic conductivity > 0.2 S cm^{-1}

Q2
- 2g stable doped metal oxide
- B.E.T. surface area > 30 m^2 g^{-1}; Electronic conductivity >0.2 S cm^{-1}

Q3
- 2g TiO_2 using SSM
- B.E.T. surface area >50 m^2 g^{-1}; Particle size <70nm

Q4
- 2g Ta-doped TiO_2 support using SSM
- B.E.T. area >50 m^2 g^{-1}; Particle size <70nm, conductivity > 0.2 S cm^{-1}

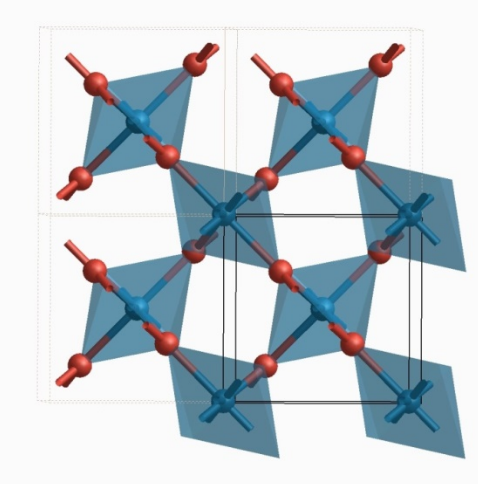
NO GO

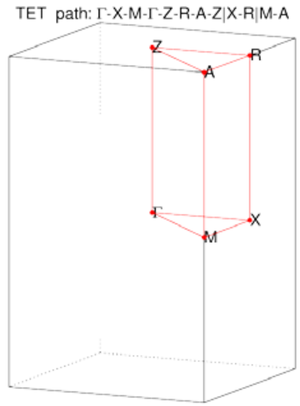
GO

Approach

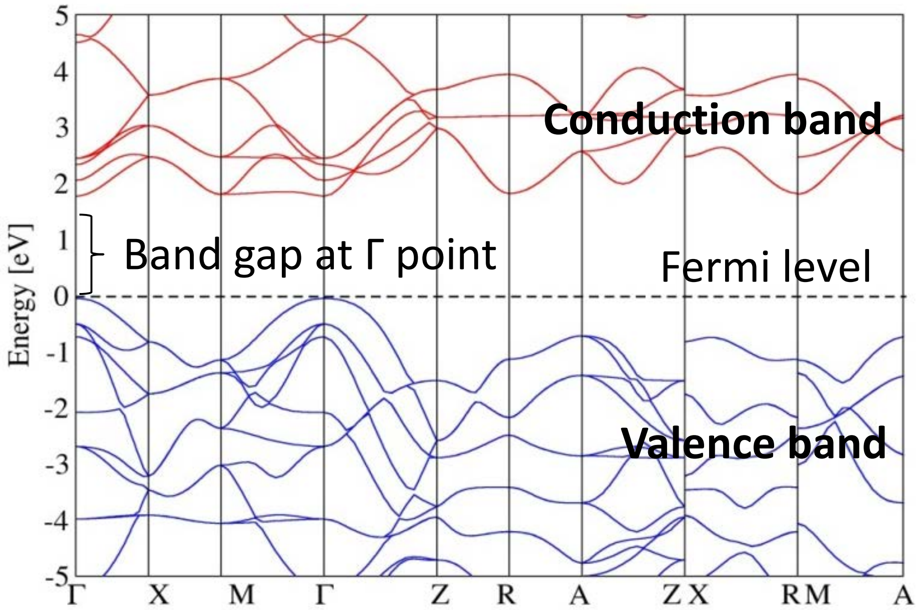
Density Functional Theory - Doping of TiO_2 with Ta

Change in the electronic structure of supports as a result of doping





DFT optimized structure of TiO_2 (PBEsol functional). Cell parameters *a*=4.56, *b*=4.56, *c*=2.93 Å
red – oxygen, blue - Ti



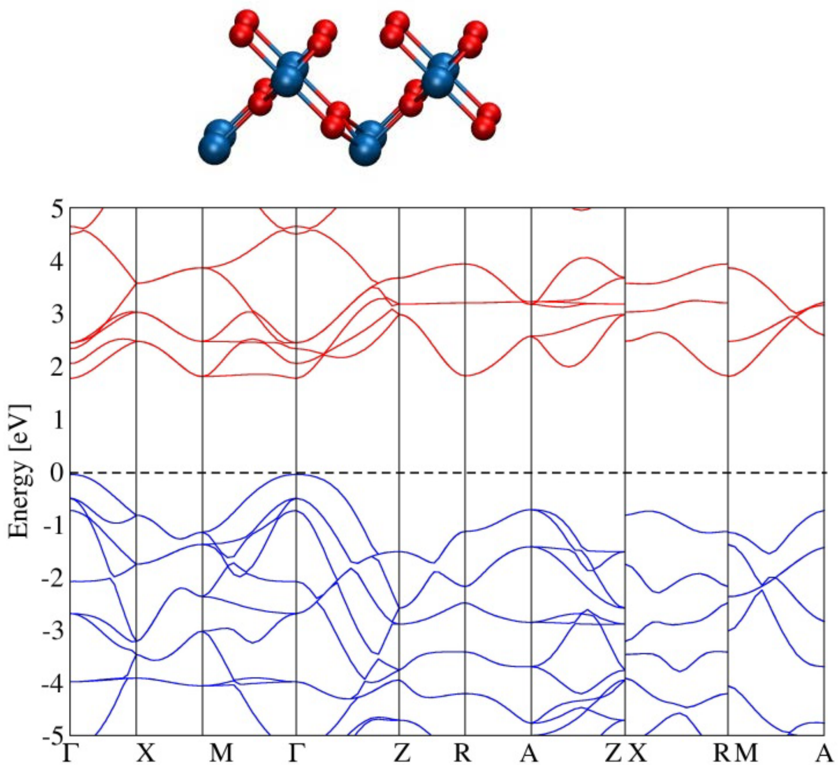
DFT calculated band structure of TiO_2. Top HSE06 level, bottom PBEsol level

- TiO_2 is **a semiconductor, absorbs in UV.**
- Direct B-G of 1.82 eV at PBEsol level, 3.44 eV at HSE06 level (hybrid functional needed).
- Experimental reports 3.3-3.6 eV (UPS-IPS spectroscopy).

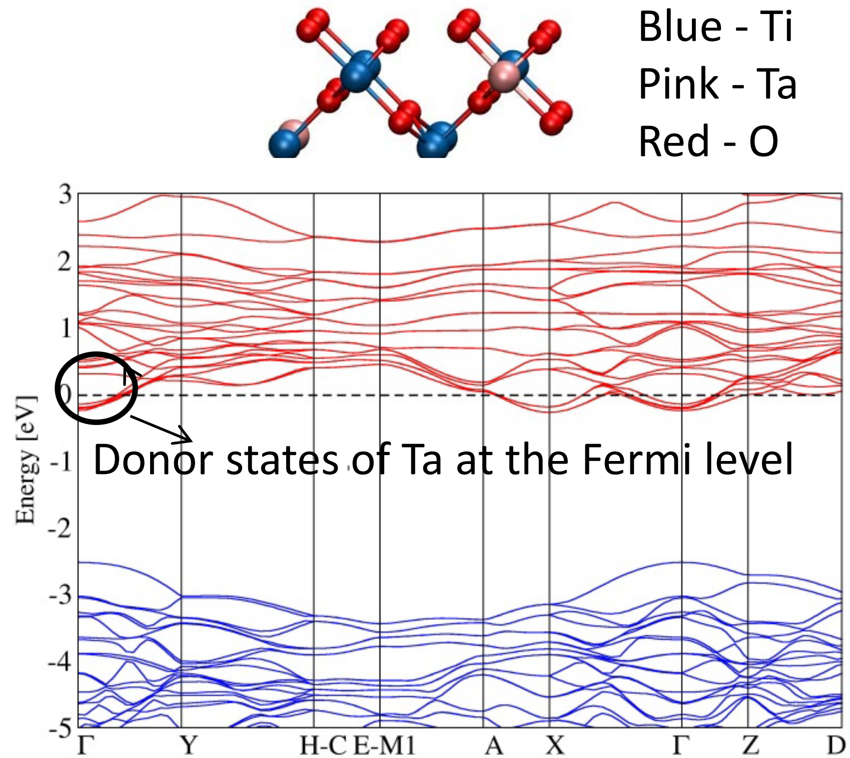
Approach

Density Functional Theory - Doping of TiO_2 with Ta

Change in the electronic structure of supports as a result of doping



TiO_2



Blue - Ti
Pink - Ta
Red - O

TiO_2 with 12.5% Ta (model concentration)

- TiO_2 is **a semiconductor**, while doping of Ta creates a ***n*-type semiconductor** with **increased conductivity** - leads to “metallization”

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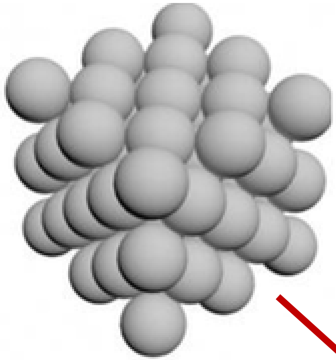
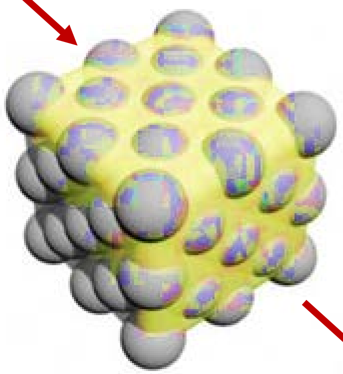
Approach

Design Porous TiO_2 supports



Silica template



Infiltration of TiO_2 support via ultra sonication, followed by pyrolysis

Leaching the sacrificial silica support: Porous TiO_2 support

Synthesis and characterization of high surface area TiO_2 supports.

(i) Synthesis of TiO_2 support.

- sol−gel technique
- alkoxides titanium as precursors

ii Sacrificial support method (Templating)

- Cab-O-Sil L90 surface area ~90 $m^2 g^{-1}$, 0.22 μm
- Cab-O-Sil EH5, surface area ~400 $m^2 g^{-1}$, 0.14 μm
- pyrolyzed at 850°C followed by leaching with 40 wt.% HF

iii Characterization of TiO_2 support

- Morphology: SEM, N_2-sorption BET surface area, pore size analysis
- Composition: EDS, XPS, Elemental Mapping
- Structure : XRD
- electron conductivity (in-house test cell)

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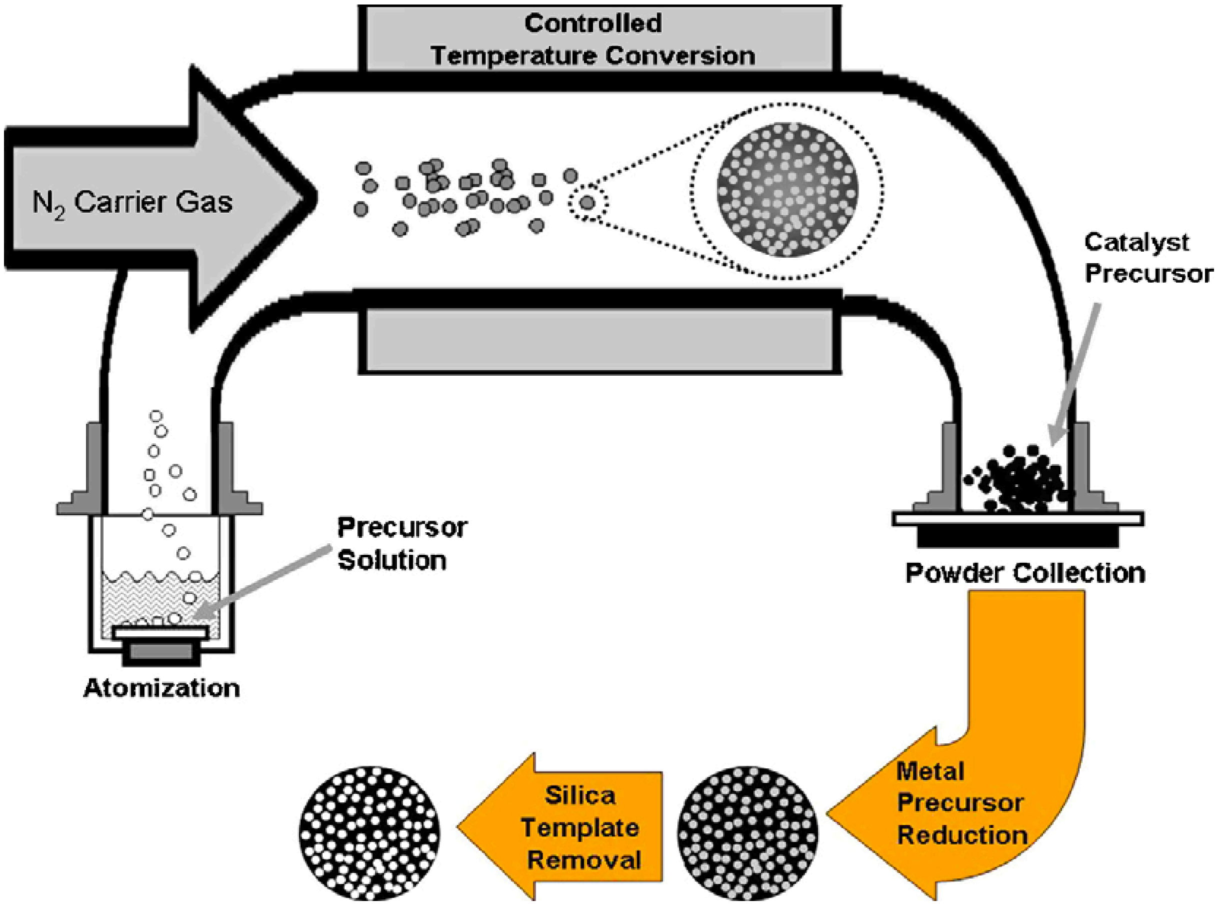
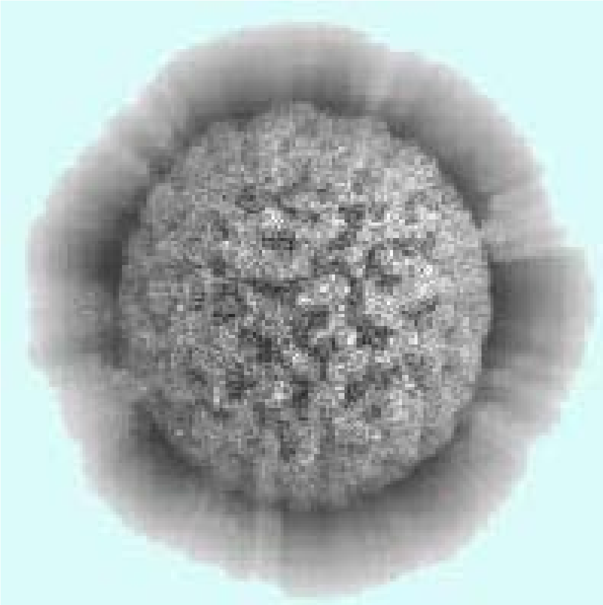
Approach

Scale-up of templated materials



Combination of spray pyrolysis with SSM method

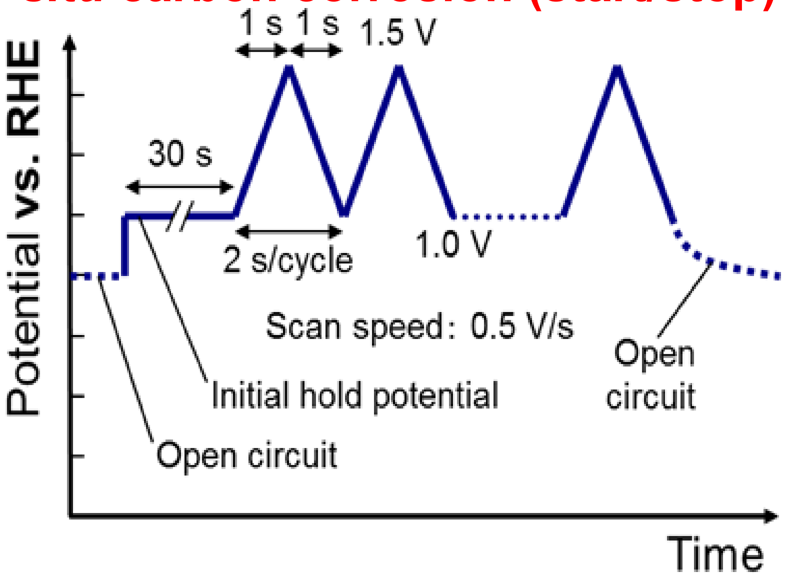


E. Switzer, P. Atanassov A.K. Datye, Nanostructured Anode Pt-Ru Electrocatalysts for Direct Methanol Fuel Cells, *Topics in Catalysis*, 46 (2007) 334-338
E. Switzer, T.S. Olson, A. K. Datye, P. Atanassov, M.R. Hibbs and C.J. Cornelius, Templated Pt-Sn Electrocatalysts for Ethanol, Methanol and CO Oxidation in Alkaline Media, *Electrochimica Acta* 54 (2009) 989-995
A. Falase, K. Garcia, C. Lau, and P. Atanassov, Electrochemical and *in Situ* IR Characterization of PtRu Catalysts for Complete Oxidation of Ethylene Glycol and Glycerol, *Electrochemistry Communications*, 13 (2011) 1488–1491

Approach

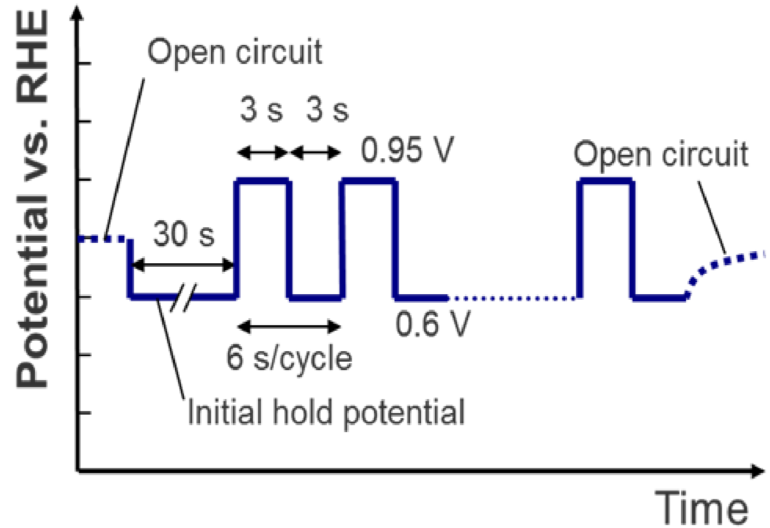
Potential cycling to evaluate support and electrocatalyst electrochemical stability/durability

Catalyst durability: *Ex-situ* and *in situ* carbon corrosion (start/stop)



Protocol for simulating start-up/shut-down phenomena

Catalyst durability: *Ex-situ* and *in-situ* Pt dissolution (load cycling)



Protocol for simulating load cycling phenomena.

The protocols recommended in solicitation **DE-FOA-0001224 (next slide)** will also be employed.

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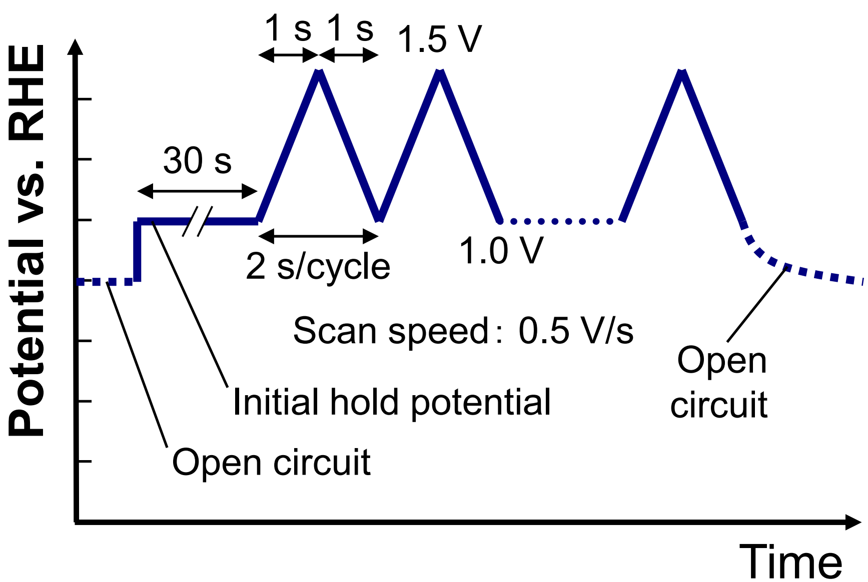
Approach

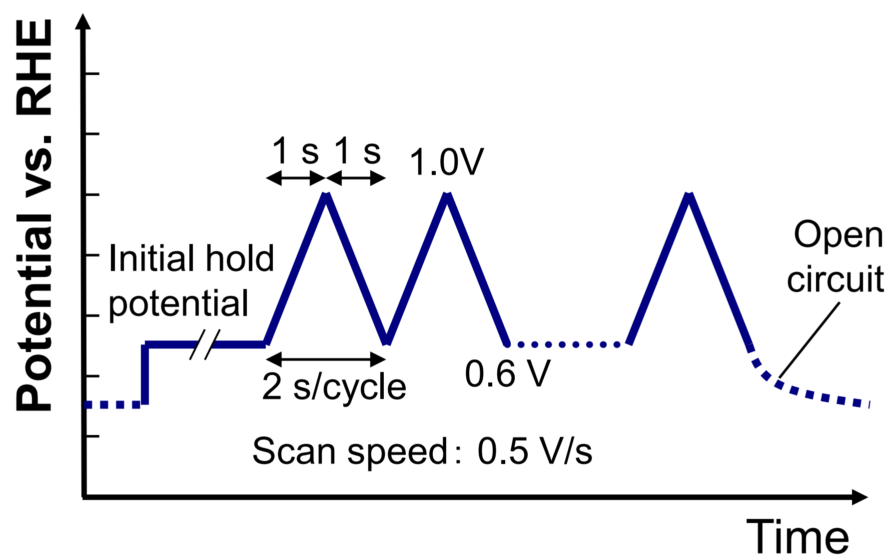
Potential cycling to evaluate support and electrocatalyst electrochemical stability/durability

Catalyst durability: *Ex-situ* and *in situ* carbon corrosion (start/stop)



Support durability — support corrosion

Catalyst durability: *Ex-situ* and *in-situ* Pt dissolution (load cycling)



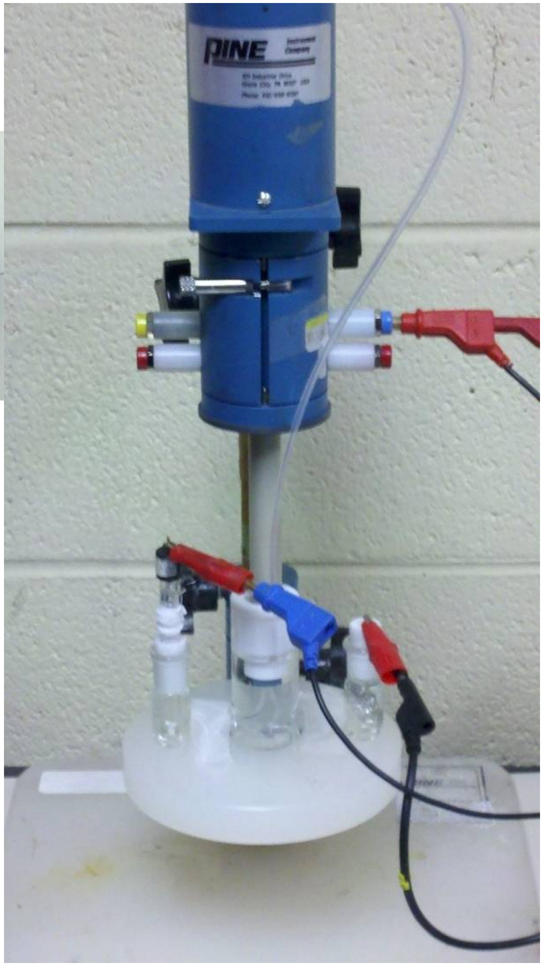
Catalyst durability – Pt dissolution

Electrolyte: 0.1 M $HClO_4$
Temperature: 60ºC at NTCNA, RT at IIT
CV sweep rate of 20 mV/s; Room temperature CV

Approach

Potential cycling to evaluate support and electrocatalyst durability

- Three electrode cell with rotating disk electrode
  - Working electrode (WE) : Glassy carbon coated with catalyst support
  - Counter electrode : Pt foil
  - Reference electrode : Saturated calomel electrode (SCE)
  - Electrolyte : N_2 saturated 0.1M $HClO_4$
- Support loading on W.E.: 200-600 $\mu g/cm^2_{geo}$ (material dependent)
- Pt loading: $20 \mu g_{Pt}/cm^2_{geo}$
- Potential cycling protocol



Approach

MEA fabrication and optimization

Homogenizer (IKA)

- NTCNA has extensive expertise in the fabrication of catalyst-coated membranes (CCMs) and catalyst-coated gas diffusion layers (GDLs).
- High performance dispersing homogenizers for uniformly dispersed catalyst ink preparation.
- Automated robotic spray system for catalyst layer deposition on GDL/membrane.

MEA fabrication

Spray system (Asymtek)

Design and optimization of catalyst layers (CL)

Estimation of ionomer volume fraction

$$\varepsilon_i \equiv \frac{V_{I,wet}}{V_{cath}} = \left(\frac{I}{C}\right)\frac{10}{f_t d_{I,dry}}\left(1 + \frac{M_w d_{I,dry}\lambda}{d_w EW}\right)$$

Estimation of ionomer film thickness

$$\sigma\ (\text{ionomer film thickness}) = \frac{V_{Nafion}}{A_{eff}} = \frac{V_{Nafion}}{(1-\gamma)A_{BET}\cdot 10^4 \cdot m_{cat}}$$

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Performance Evaluation

MEA conditions (electrochemical diagnostics)		
Temperature		80 °C
Anode	Gas	H_2
	Relative humidity	100%
	Flow rate (NLPM)	0.5
Cathode	Gas	N_2
	Relative humidity	100%
	Flow rate (NLPM)	0.5

MEA conditions (iV performance)	
BoL and EoL iV performance	H_2-O_2/Air, 80°C, RH 40%, RH 100%, ambient pressure, and 101 kPa (gauge pressure)

- Fuel cell performance evaluation under standard DOE-protocols
- To better understand mass transport properties.
- Using dilute oxygen concentrations (~0.5-2% O_2) to obtain gas transport resistances (R_{diff} and R_{other}) in the catalyst layer.

Approach

1st year milestones and GNG

Task number	Milestone	Milestone description	Milestone verification process	Anticipated Date/Quarter	Current status
1	Milestone 1.1	2g of TiO_2-Ta*	B.E.T. surface area >30 $m^2 g^{-1}$; electronic conductivity > 0.2 S cm^{-1}	M3/Q1	20 $m^2 g^{-1}$; 0.1 S cm^{-1}
4	Milestone 4.1	2g of stable doped-metal-oxide support	B.E.T. surface area > 30 m^2 g^{-1}; electronic conductivity >0.2 S cm^{-1}	M6/Q2	Not started
5	Milestone 5.1.1	2g of TiO_2 using SSM	B.E.T. area >50 m^2 g^{-1}; particle size <70nm	M9/Q3	Not started
5	Milestone 5.1.2 Go/No-Go	2g of TiO_2-Ta support material using SSM	B.E.T. area >50 m^2 g^{-1}; particle size <70nm, conductivity > 0.2 S cm^{-1}	M12/Q4	Not started

* Or any other conducting and stable doped-metal-oxide support exhibiting SMSI and meeting the milestone targets

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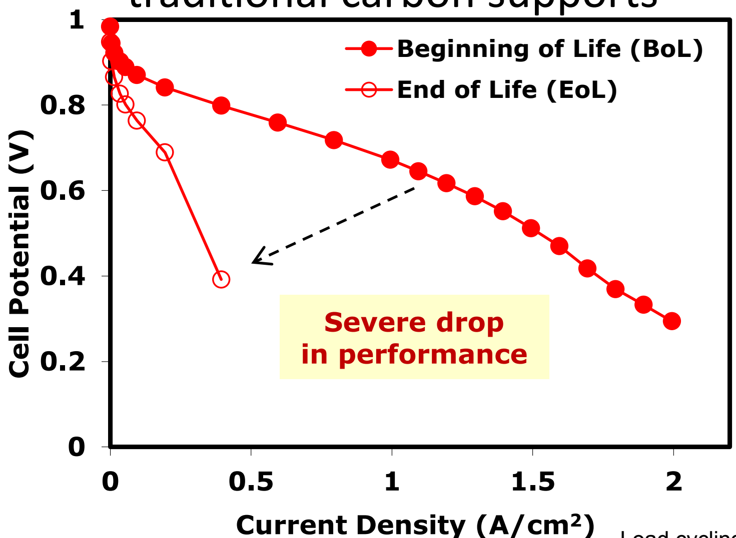
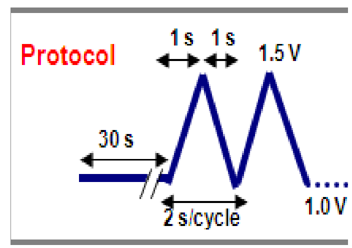
Technical accomplishments

IIT-Nissan Pt/non-carbon support research: Example of previous results

- Problem: Poor durability of traditional carbon supports



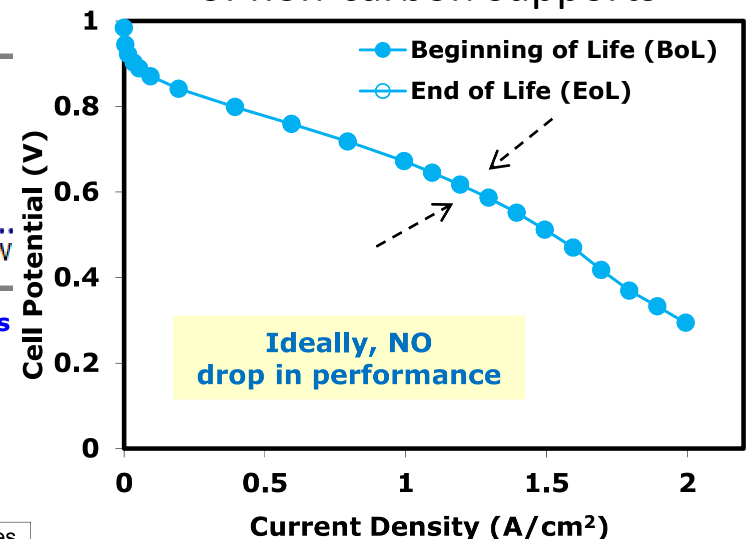
FCCJ (Japan)



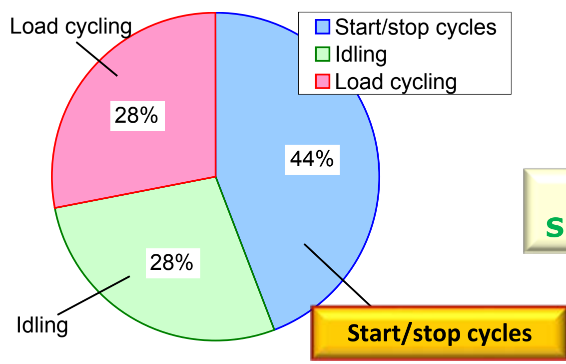
1000 cycles

Start-stop potential cycling protocol

- Approach: Development of of non-carbon supports

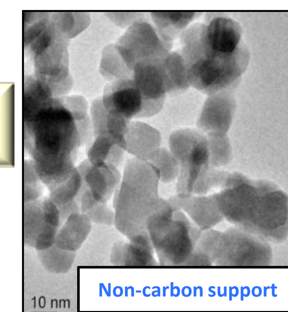


Nissan study on fuel cell degradation modes



Shimoi et al, JSAE Spring Meeting (2009)

Example: TiO_2-RuO_2, SnO_2-In_2O_3 metal oxides



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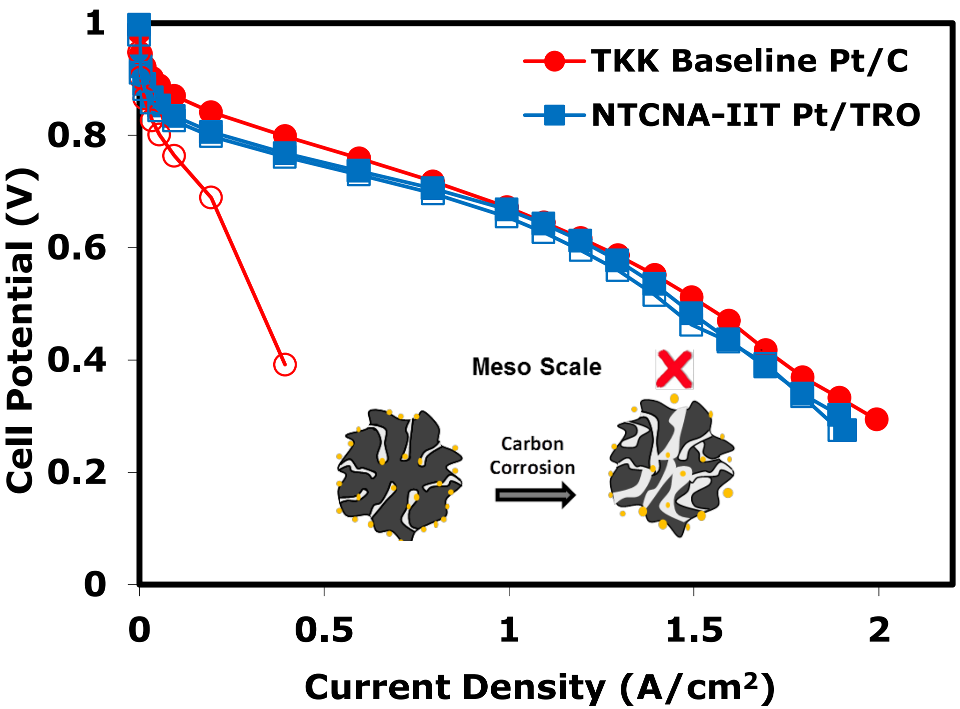
Technical accomplishments

Pt/TRO as cathode support – *Prior DOE Project*

- Pt/TRO showed excellent durability under start-stop protocol



Durability of Pt/TRO is much better than the Pt/C baseline catalyst

- Published in PNAS*

Breakthrough durability of Pt/TRO Results published in PNAS

- * Illustrative cover only!

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Technical accomplishments

Start-stop stability of Ta doped TiO_2 in a PEFC

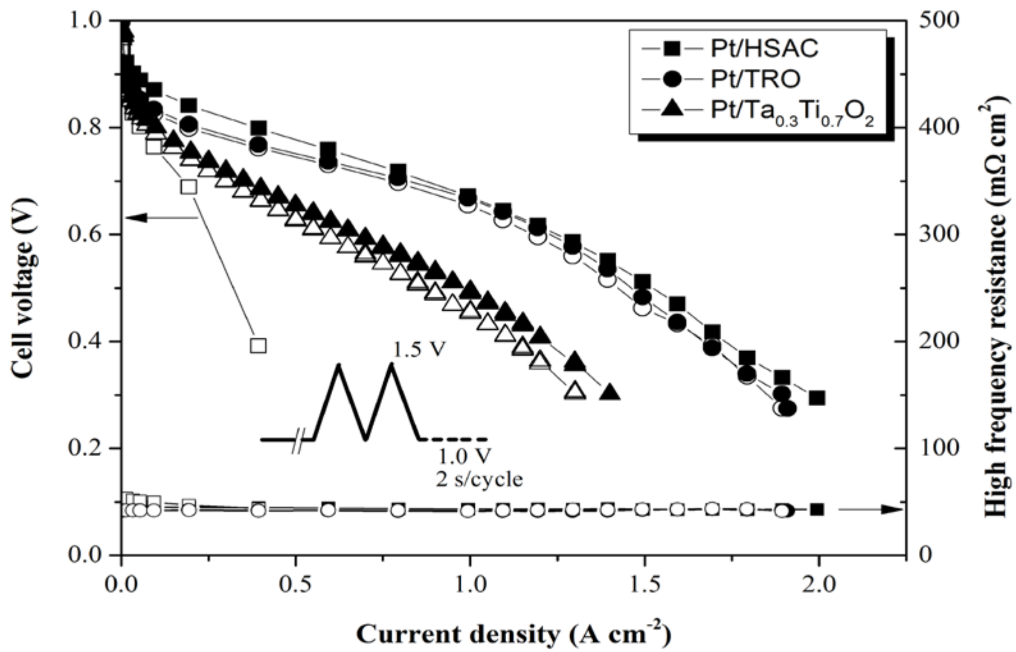

Figure . Comparison of fuel cell performance obtained with Pt/ HSAC, Pt/TRO, and Pt/ (TiO_2-Ta) before (closed symbols) and after (open symbols) exposure to the start–stop protocol specified in FOA (1,000 cycles). 25 cm² fuel cell; 80ºC and 100% RH.

- Pt/TiO_2-Ta shows remarkable start-stop stability
- Ability to achieve respectable performance (though short of Pt/TRO or Pt/C) with *essentially zero optimization* of the Pt/TiO_2-Ta electrode.

Note: TRO is TiO_2-RuO_2, developed in our previous project

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Demonstration of SMSI in Ta doped TiO_2

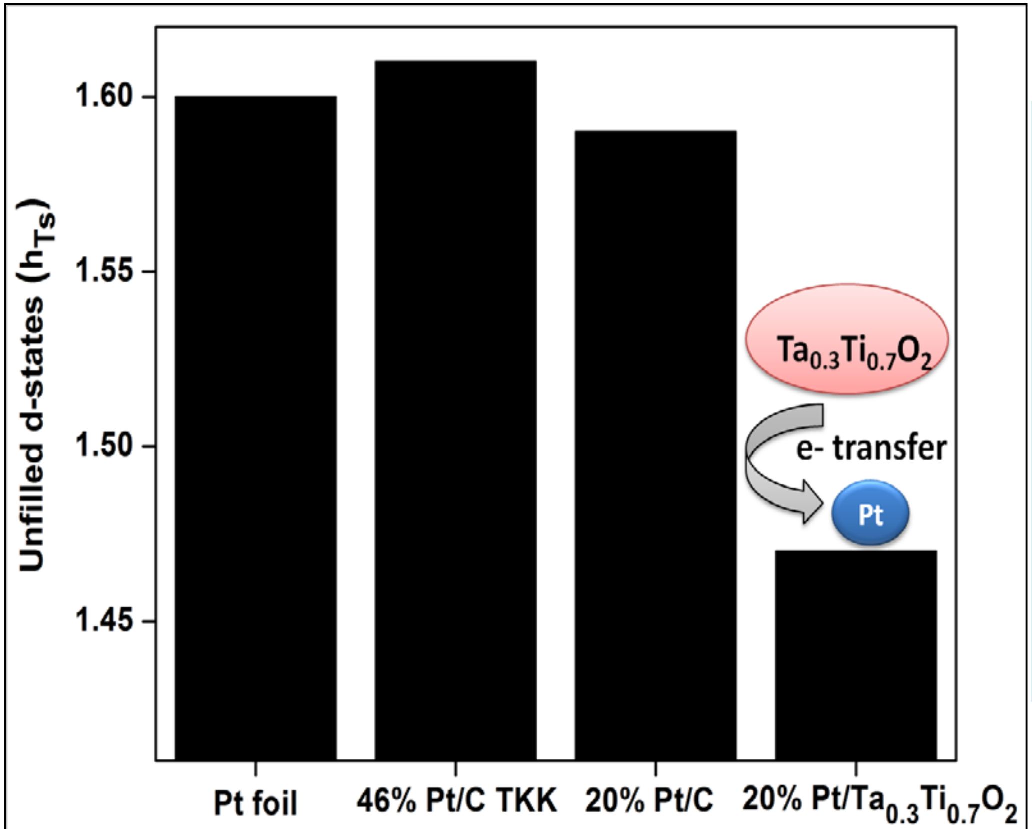


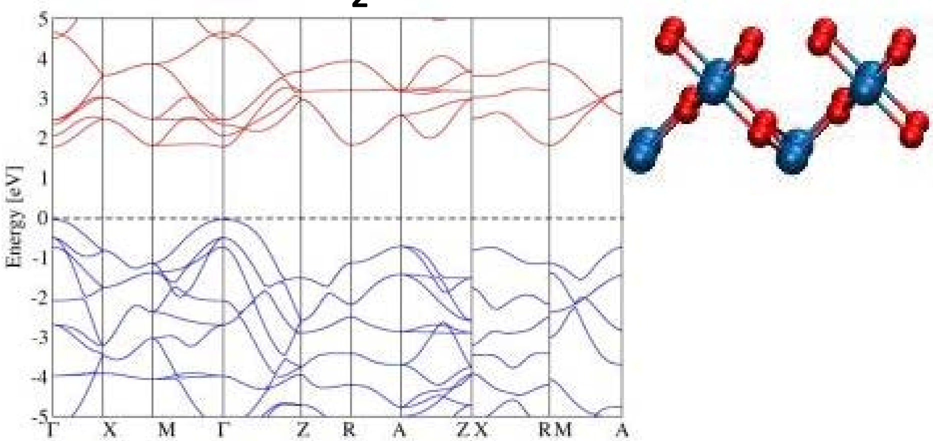
Figure. Variation in unfilled d-states for 20% Pt/TiO_2-Ta, 46% Pt/C, 20% Pt/C catalysts and Pt foil.

- The existence of SMSI on Pt/TiO_2-Ta was ascertained by XPS and XAS.
- The decrease in the number of unfilled d-states confirms electron donation from the TiO_2-Ta support to Pt nanoparticles.
- SMSI mitigates Pt dissolution under load cycling conditions

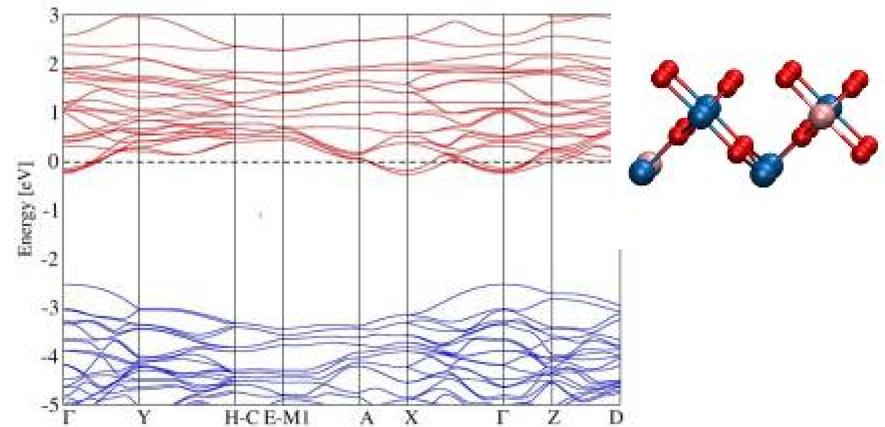
Technical accomplishments

DFT calculations for Ta-TiO_2 support

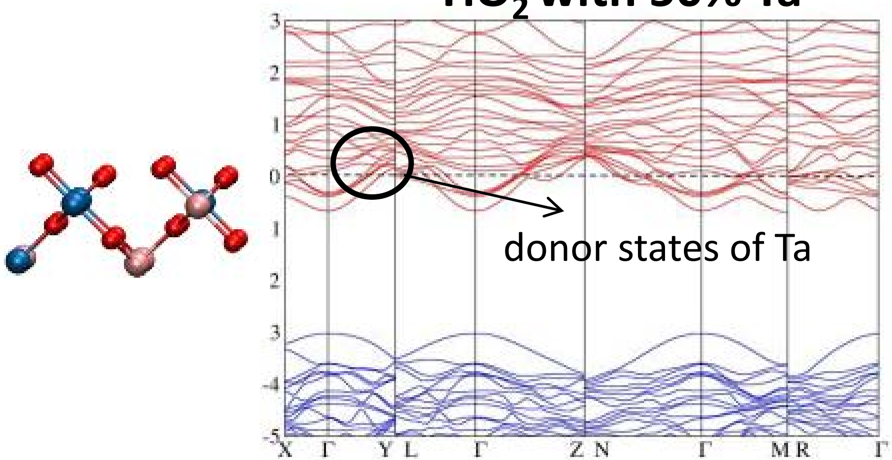
TiO_2



TiO_2 with 25% Ta



TiO_2 with 50% Ta



- DFT calculations show that doping TiO_2 with Ta from 25-50% reduces the B-G
- Ta-TiO_2 becomes increasingly metallic and conductive.

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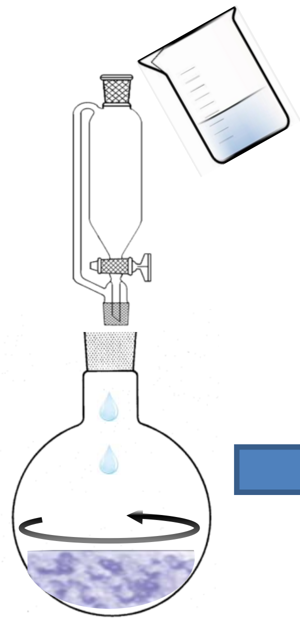
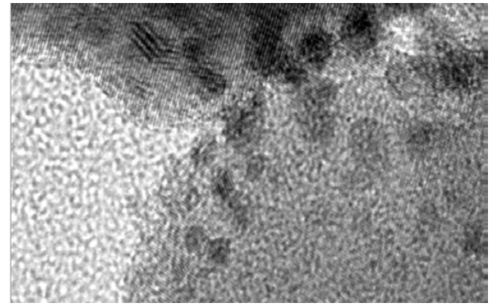
Technical accomplishments

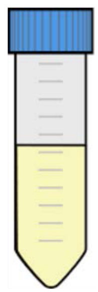
Sol-gel synthesis

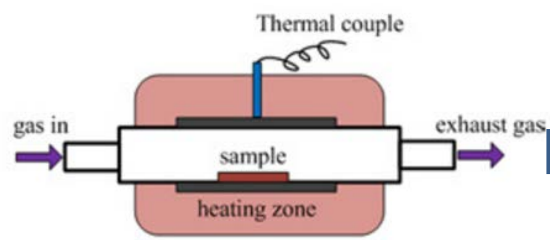


Precursors A+B in ethanol

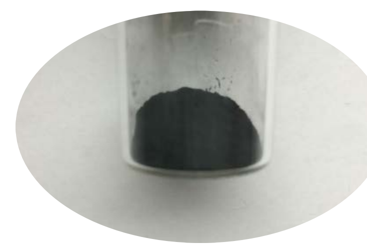


Pt deposition
(formic acid reduction)





Catalyst support



Water+ ethanol
Inert atmosphere

Centrifuge and wash the material

Anneal under 4% H_2 at T=200-1200°C

- Precursors: Metal alkoxides
- High water/ethanol to alkoxide ratio
- B.E.T surface area: 10- 20 m^2/g

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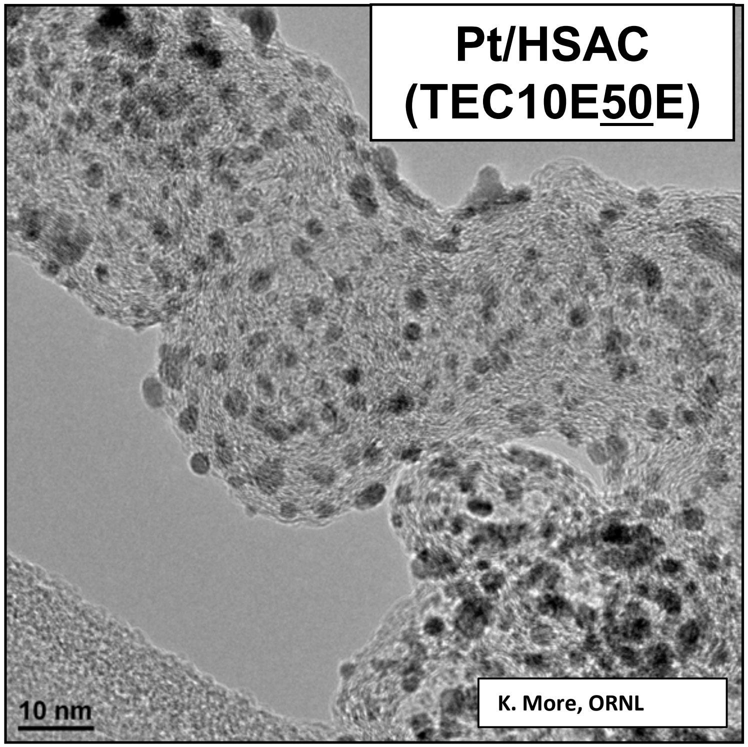
Technical accomplishments

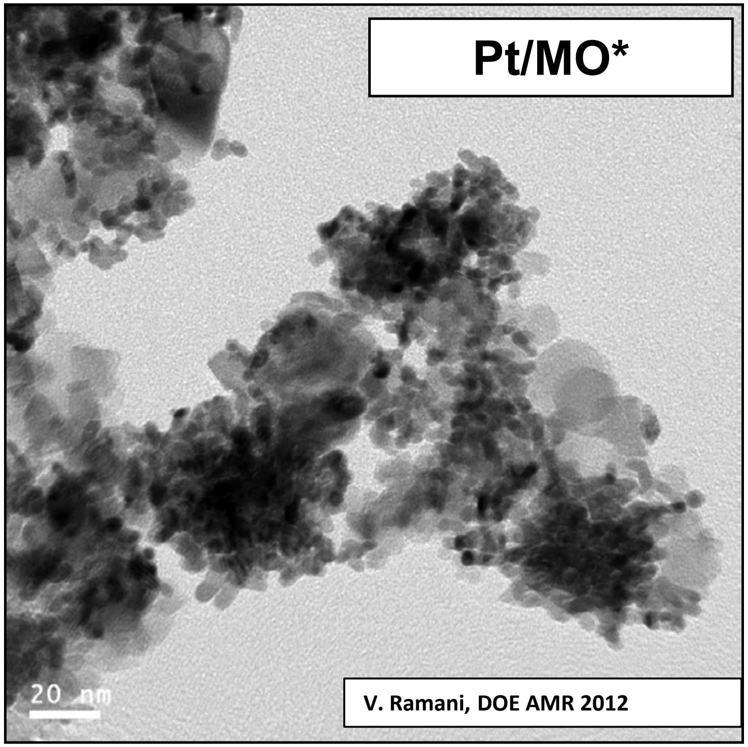
Sol-gel synthesis

Oxide	Precursor A	Precursor B	Annealing temperature	BET m^2/g	Conductivity S/cm
Nb-doped TiO_2	$Nb_2(OC_2H_5)_{10}$	$Ti[OCH(CH_3)_2]_4$	650°C	14.6 ± 2.4	0.102 ± 0.01
Nb-doped TiO_2	$Nb_2(OC_2H_5)_{10}$	$Ti[OCH(CH_3)_2]_4$	550°C	21.2 ± 3.1	Non Conductive
Ta-doped TiO_2	$Ta_2(OC_2H_5)_{10}$	$Ti[OCH(CH_3)_2]_4$	1000°C	3.4 ± 0.5	0.043 ± 0.007
Ta-doped TiO_2	$Ta_2(OC_2H_5)_{10}$	$Ti[OCH(CH_3)_2]_4$	850°C	10.3 ± 1.3	0.024 ± 0.005
Ta-doped Nb_2O_5	$Ta_2(OC_2H_5)_{10}$	$Nb_2(OC_2H_5)_{10}$	850°C	12.1 ± 1.7	Non Conductive

Remaining Challenges and Barriers

TEM images of Pt/C and Pt/MO*





- ❑ There are significant differences between Pt/C and Pt/MO
- ❑ Pt particle size, Pt dispersion/agglomeration, Pt particle density.
- ❑ Engineer wettability

* MO= metal oxides

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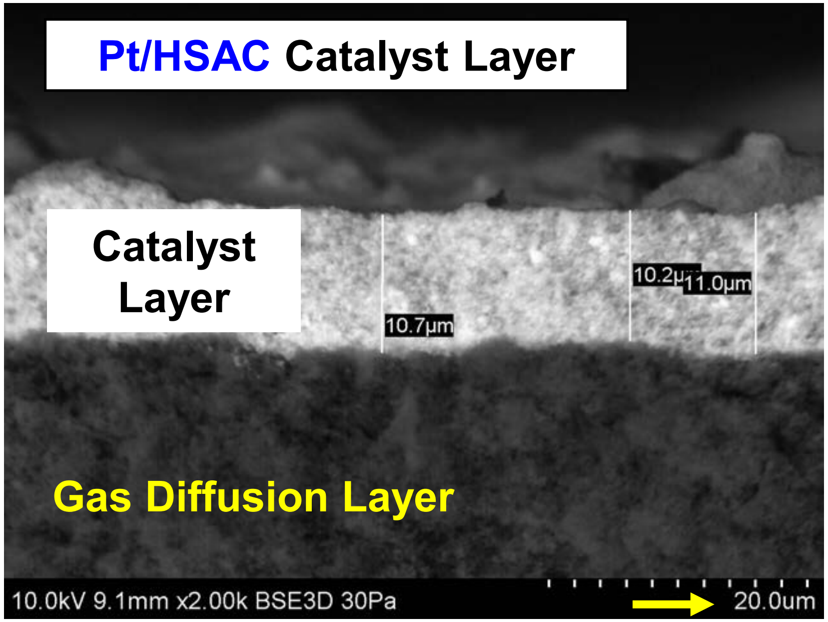
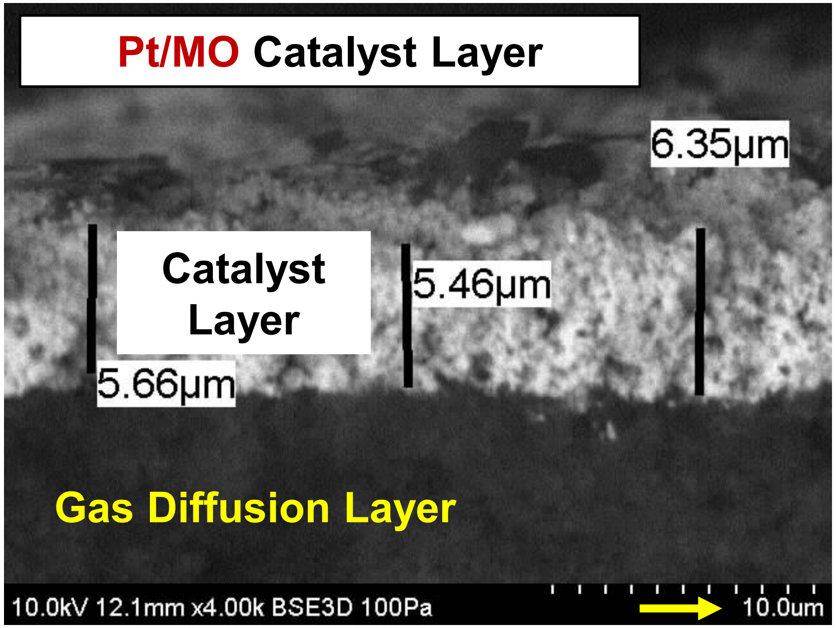
Remaining Challenges and Barriers

SEM pictures of Pt/C and Pt/MO* catalyst layers





	Pt/HSAC	Pt/MO
CL thickness (μm)	11	**5.5**
I/C mass ratio	0.9	0.9
B.E.T. surface area(m^2/g)	313	**39**
ε_i (ionomer volume fraction)	0.21	**0.66**

$$\varepsilon_{\rm i} \equiv \frac{V_{\rm I,wet}}{V_{\rm cath}} = \left(\frac{I}{C}\right)\frac{10}{f_{\rm t}d_{\rm I,dry}}\left(1 + \frac{M_{\rm w}d_{\rm I,dry}\lambda}{d_{\rm w}{\rm EW}}\right)$$

- ❑ MO is denser than carbon
- ❑ The Pt/MO CL is much thinner than Pt/HSAC.
- ❑ The ionomer volume fraction (ε_i) is higher in Pt/MO
- ❑ Optimize MEA composition and design

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Remaining Challenges and Barriers

Project just started!!!

Task number	Milestone	Milestone description	Milestone verification process	Anticipated Date/Quarter
1	Milestone 1.1	2g of TiO_2-Ta*	B.E.T. surface area >30 m^2g^{-1}; electronic conductivity > 0.2 S cm^{-1}	M3/Q1
4	Milestone 4.1	2g of stable doped-metal-oxide support	B.E.T. surface area > 30 m^2 g^{-1}; electronic conductivity >0.2 S cm^{-1}	M6/Q2
5	Milestone 5.1.1	2g of TiO_2 using SSM	B.E.T. area >50 m^2 g^{-1}; particle size <70nm	M9/Q3
5	Milestone 5.1.2 Go/No-Go	2g of TiO_2-Ta support material using SSM	B.E.T. area >50 m^2 g^{-1}; particle size <70nm, conductivity > 0.2 S cm^{-1}	M12/Q4

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Remaining Challenges and Barriers

Task Number	Milestone	Milestone Description	Milestone Verification Process*	Anticipated Date/Quarter
7	Milestone 7.1	2g of Pt/DS catalyst (SMSI)	Demonstrate SMSI; Meets Table 2 durability targets in RDE	M15/Q5
8	Milestone 8.1	Pt/DS catalyst	Demonstrate 10% increase in mass activity	M18/Q6
5	Milestone 5.2.1	2g of at least one doped oxide using SSM	B.E.T. area >70 m^2g^{-1}; particle size <70nm; conductivity ; > 0.2 Scm^{-1}; Stability and durability in RDE per DOE metrics	M21/Q7
6	Milestone 6.2.1 Go/No-Go	Deliver 2g of Pt/DS catalyst to NTCNA	20-40wt%Pt; > 70 m^2g^{-1}; Pt particle size 3-6nm; meets DOE 2020 durability targets	M24/Q8

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Remaining Challenges and Barriers

Task Number	Milestone	Milestone Description	Milestone Verification Process*	Anticipated Date/Quarter
10	Milestone 10.1	Pt/DS catalyst	Demonstrate "End Project" durability metrics and at least 80% of mass activity metric	M27/Q9
6	Milestone 6.2.2	Pt/DS catalyst	In addition to Milestone 6.2.1, meet "End Project" BoL mass activity target	M30/Q10
11	Milestone 11.1	Deliver cost model	Specify cost of best 2 Pt/DS materials	M33/Q11
12	Milestone 12.1 Go/No-Go	Deliver six 50 cm^2 active area MEAs to DOE	Meet "End Project" durability, activity, and performance targets in Table 2	M36/Q12

Collaboration

Illinois Institute of Technology

- *Lead PI and Technical PoC*: **Vijay K. Ramani**
- Metal oxide synthesis and characterization, RDE testing (ORR activity and electrochemical stability), PEFC diagnostics

Nissan Technical Center, North America

- PI and Technical PoC: **Nilesh Dale**
- Evaluation of the catalysts in RDE and PEFC, Cost modeling

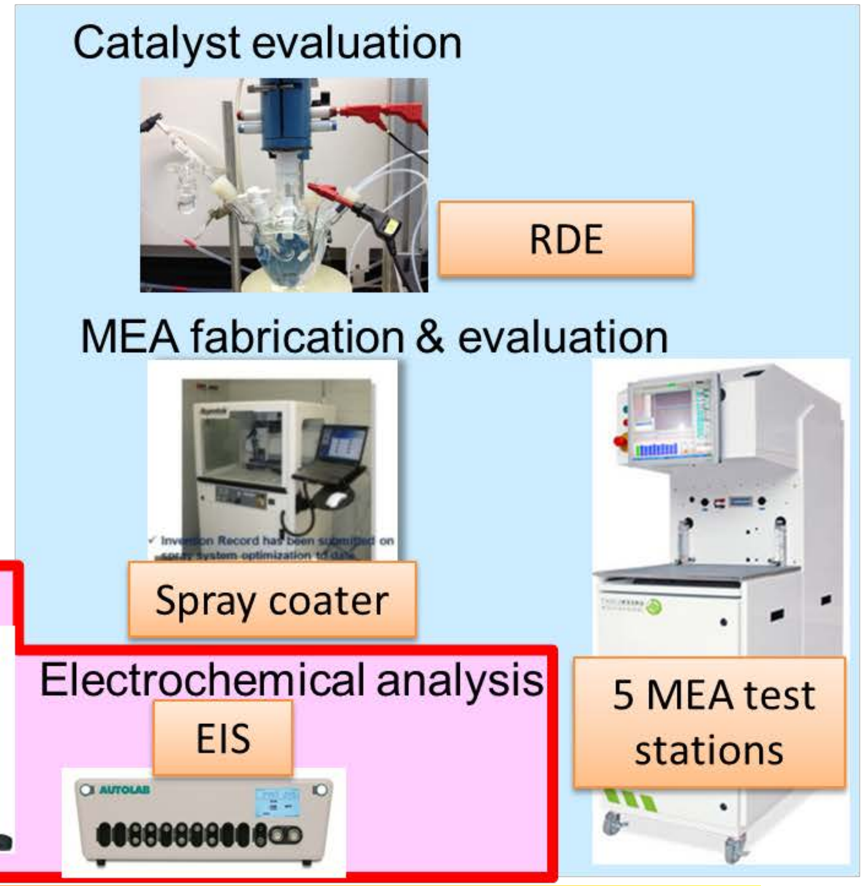
University of New Mexico

- *PI and Technical PoC*: **Plamen Atanassov**
- Modeling of doped MO conductivity and SMSI (DFT), scale-up of doped metal oxide synthesis

Collaboration

Facility and Equipment Capabilities

- Scanning Electron Microscope (SEM, EDS)
- X-ray Fluorescence spectrometer (XRF): To determine the Pt loading.
- 5 fuel cell test test-stations (Hydrogenics)
- Expertise in the fabrication and characterization of catalyst layer (CL): ionomer volume fraction, proton transport resistance, and oxygen transport resistance.
- Rotating Disk Electrode: *ex-situ* catalyst performance and durability



Proposed Future Work

FY 2016

- IIT: materials synthesis and characterization
  - ✓ Synthesis and characterization of Ta doped TiO_2 and other doped metal oxides using wet chemistry
  - ✓ Electrochemical evaluation of support and Pt/MO stability
  - ✓ Investigation of SMSI in Pt/doped-metal-oxide systems
  - ✓ Measurement of BoL ECSA and ORR activity of selected catalysts

- Nissan North America Inc.: durability/performance testing
  - ✓ Accelerated test protocols on materials provided by IIT
  - ✓ Fabrication / testing of sub-scale and 50 cm^2 MEAs

- University of New Mexico
  - ✓ DFT calculations: conductivity and SMSI of relevant doped metal oxides
  - ✓ Characterization of the doped metal oxides and derived catalysts
  - ✓ High surface area support synthesis by SSM.

Summary

- **Objectives and Approach:**
  - Synthesize doped metal oxides for catalyst supports
  - High conductivity and BET surface area
  - Exhibits SMSI and corrosion resistant (attaining DOE 2020 targets)
- **Relevance**
  - Material-level mitigation strategies can solve cathode durability issues
- **Accomplishments**
  - DFT framework in place to study effect of doping on conductivity
  - Successfully synthesized doped metal oxides with conductivities of 0.1 S/cm
- **Collaborations**
  - Illinois Institute of Technology
  - Nissan Technical Center, North America
  - University of New Mexico

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