



• In situ studies of methaol fuel cells



- In situ studies of methaol fuel cells
- Reversibility in tin-based anode materials



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- Reversibility in tin-based anode materials
- Angle Resolved Photoemission



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- HAXPES



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Reading Assignment: Chapter 8.1–8.3



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Homework Assignment #06: Chapter 6: 1,6,7,8,9 due Tuesday, November 16, 2021



- In situ studies of methaol fuel cells
- Reversibility in tin-based anode materials
- Angle Resolved Photoemission
- HAXPES

Reading Assignment: Chapter 8.1–8.3

Homework Assignment #06: Chapter 6: 1,6,7,8,9 due Tuesday, November 16, 2021 Homework Assignment #07: Chapter 7: 2,3,9,10,11 due Tuesday, November 30, 2021





R. Viswanathan et al., "In-situ XANES study of carbon supported Pt-Ru anode electrocatalysts for reformate-air polymer electrolyte fuel cells," J. Phys. Chem. B 106, 3458 (2002).

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• Transmission mode



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• Transmission mode

• <1 mm of graphite

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- Pt/Ru on anode



- <1 mm of graphite
- Pd on cathode

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- Transmission mode
- Pt/Ru on anode
- 35°C operating temp



- <1 mm of graphite
- Pd on cathode

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- Transmission mode
- Pt/Ru on anode
- 35°C operating temp



- <1 mm of graphite
- Pd on cathode
- 1-2 min scan time

R. Viswanathan et al., "In-situ XANES study of carbon supported Pt-Ru anode electrocatalysts for reformate-air polymer electrolyte fuel cells," J. Phys. Chem. B 106, 3458 (2002).

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E.A. Lewis et al., "Operando x-ray absorption and infrared fuel cell spectroscopy", Electrochim. Acta. 56, 8827 (2011).

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• Air-breathing cathode

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• Air-breathing cathode

Pd on anode

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- Air-breathing cathode
- 1.2 mg/cm^2 loading

Pd on anode

E.A. Lewis et al., "Operando x-ray absorption and infrared fuel cell spectroscopy", Electrochim. Acta. 56, 8827 (2011).

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- Air-breathing cathode
- 1.2 mg/cm^2 loading



- Pd on anode
- 50°C operating temp

E.A. Lewis et al., "Operando x-ray absorption and infrared fuel cell spectroscopy", Electrochim. Acta. 56, 8827 (2011).

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- Air-breathing cathode
- 1.2 mg/cm^2 loading
- Pt L₃ and Ni K edges



- Pd on anode
- 50°C operating temp

E.A. Lewis et al., "Operando x-ray absorption and infrared fuel cell spectroscopy", Electrochim. Acta. 56, 8827 (2011).

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- Air-breathing cathode
- 1.2 mg/cm^2 loading
- Pt L₃ and Ni K edges



- Pd on anode
- 50°C operating temp
- Continuous scan mode

E.A. Lewis et al., "Operando x-ray absorption and infrared fuel cell spectroscopy", Electrochim. Acta. 56, 8827 (2011).

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U.S. Department of Defense (DoD) Fuel Cell Test and Evaluation Center (FCTec)



Anode: 0 V vs. SHE $2 H_2 \longrightarrow 4 H^+ + 4 e^-$



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Anode: 0 V vs. SHE $2 H_2 \longrightarrow 4 H^+ + 4 e^-$

Cathode: 1.23 V vs. SHE

$$O_2 + 4 H^+ + 4 e^- \longrightarrow 2 H_2O$$



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U.S. Department of Defense (DoD) Fuel Cell Test and Evaluation Center (FCTec)



Anode: 0 V vs. SHE $2 H_2 \longrightarrow 4 H^+ + 4 e^-$

Cathode: 1.23 V vs. SHE $O_2 + 4 H^+ + 4 e^- \longrightarrow 2 H_2O$

breaking $O{-}O$ bond is the rate limiting step



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PtNi/Pd has higher open circuit voltage, similar performance to Pt/Pd.

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PtNi/Pd has higher open circuit voltage, similar performance to Pt/Pd.





PtNi/Pd has higher open circuit voltage, similar performance to Pt/Pd.

Why is ORR improved with bimetallic catalyst?

• Pt electronic structure modified





PtNi/Pd has higher open circuit voltage, similar performance to Pt/Pd.

- Pt electronic structure modified
- Pt catalyst geometric structure modified



PtNi/Pd has higher open circuit voltage, similar performance to Pt/Pd.

- Pt electronic structure modified
- Pt catalyst geometric structure modified
- Static oxygen adsorbates inhibited



PtNi/Pd has higher open circuit voltage, similar performance to Pt/Pd.

- Pt electronic structure modified
- Pt catalyst geometric structure modified
- Static oxygen adsorbates inhibited
- Overpotential reduced



PtNi/Pd has higher open circuit voltage, similar performance to Pt/Pd.

Why is ORR improved with bimetallic catalyst?

- Pt electronic structure modified
- Pt catalyst geometric structure modified
- Static oxygen adsorbates inhibited
- Overpotential reduced

Using XAS to study the catalyst nanoparticles can help answer the first three questions

$\ensuremath{\mathsf{Pt}/\mathsf{C}}$ and $\ensuremath{\mathsf{PtNi}/\mathsf{C}}$ comparison





Q. Jia et al, "In Situ XAFS studies of the oxygen reduction reaction on carbon supported Pt and PtNi(1:1) catalysts", *J. Phys. Conf. Series* **190**, 012157 (2009).

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Pt/C and PtNi/C comparison





At highest potential, all catalysts are metallic and the Pt L_3 XANES shows significant difference between the Pt and PtNi catalysts

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Pt/C and PtNi/C comparison





At highest potential, all catalysts are metallic and the Pt L_3 XANES shows significant difference between the Pt and PtNi catalysts

Delta XANES shows a supression of Pt oxidation in the PtNi catalyst as a function of applied potential

Q. Jia et al, "In Situ XAFS studies of the oxygen reduction reaction on carbon supported Pt and PtNi(1:1) catalysts", J. Phys. Conf. Series 190, 012157 (2009).

PtNi structural model



Attempt to get global information about the oxygen



Fit all potentials with same metal core parameters for each catalyst



Fit all potentials with same metal core parameters for each catalyst

Simultaneous fit of Pt and Ni edges in PtNi/C with constraint on Pt-Ni distance



Fit all potentials with same metal core parameters for each catalyst

Simultaneous fit of Pt and Ni edges in PtNi/C with constraint on Pt-Ni distance

Fit in k, k^2 , and k^3 weighting simultaneously



Fit all potentials with same metal core parameters for each catalyst

Simultaneous fit of Pt and Ni edges in PtNi/C with constraint on Pt-Ni distance

Fit in k, k^2 , and k^3 weighting simultaneously

Apply M-O path constraints

- length common across potentials
- σ^2 fixed to 0.01
- Pt-O in PtNi/C at all potentials are refined with a common occupation #

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"In Situ XAFS studies of the oxygen reduction reaction on carbon supported Pt and PtNi(1:1) catalysts", Q. Jia, E.A. Lewis, E.S. Smotkin, and C.U. Segre, *J. Phys. Conf. Series* **190**, 012157 (2009).

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Fits out to 3.5 Å $\,$ in R-space and back Fourier Transforms

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Fits out to 3.5 Å $\,$ in R-space and back Fourier Transforms

The Pt catalyst shows a larger oxygen path contribution than the PtNi catalyst

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8/37



Fits out to 3.5 Å $\,$ in R-space and back Fourier Transforms

The Pt catalyst shows a larger oxygen path contribution than the PtNi catalyst

The Ni EXAFS is dominated by the presence of a significant Ni-O bond

"In Situ XAFS studies of the oxygen reduction reaction on carbon supported Pt and PtNi(1:1) catalysts", Q. Jia, E.A. Lewis, E.S. Smotkin, and C.U. Segre, *J. Phys. Conf. Series* **190**, 012157 (2009).

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Fit results



Pt/C		PtNi/C			
		Pt		Ni	
N_{Pt}	8.7 ± 0.2	N_{Pt}	6.1 ± 0.3	N _{Ni}	3.7 ± 0.2
R_{Pt-Pt}	2.749 ± 0.001	R_{Pt-Pt}	2.692 ± 0.003	R _{Ni-Ni}	2.572 ± 0.006
		N _{Ni}	$\textbf{3.4} \pm \textbf{0.1}$	N_{Pt}	8.9 ± 0.5
			R _{Pt-Ni}	2.635 ± 0.004	
		N_{Total}	9.5 ± 0.4	N_{Total}	12.6 ± 0.7
R_{Pt-O}	2.02 ± 0.01	R_{Pt-O}	2.09 ± 0.03	R _{Ni-O}	1.90 ± 0.01

Note the Pt-Pt and Pt-O bond lengths as well as total metal near neighbors

"In Situ XAFS studies of the oxygen reduction reaction on carbon supported Pt and PtNi(1:1) catalysts", Q. Jia, E.A. Lewis, E.S. Smotkin, and C.U. Segre, J. Phys. Conf. Series 190, 012157 (2009).

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Role of Ru in CO oxidation?



- PtRu bifunctional catalyst improves performance
- In commercial PtRu catalysts there is always a lot of inactive Ru-oxide (?)
- Ru signal dominated by metallic Ru environment
- How does Ru behave in the presence of reactants adsorbed on platinum surface?

Core-shell nanoparticles can resolve these questions

Ru-decorated Pt nanoparticles





ANL-EMC 10.0kV 11.7mm x9.01k SE(M) 9/14/2011 5.00um









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Electrochemical performance





Without Methanol Low V peaks are H^+ stripping Dip at ~0.5 V is oxygen stripping Ru shifts potential on all peaks

"In situ Ru K-Edge x-ray absorption spectroscopy study of methanol oxidation mechanisms on model submonolayer Ru on Pt nanoparticle electrocatalyst," C.J. Pelliccione, E.V. Timofeeva, J.P. Katsoudas, and C.U. Segre, *J. Phys. Chem.* **C 117**, 18904 (2013).

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Electrochemical performance



12/37



Without Methanol Low V peaks are H^+ stripping Dip at $\sim 0.5 V$ is oxygen stripping Ru shifts potential on all peaks

With Methanol

Continual current growth is due to methanol oxidation

Ru improves current by removing the CO which blocks active sites

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Ru EXAFS



No methanol



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Fit example: -225 mV without methanol



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Fit example: 675 mV without methanol



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Fit example: 675 mV with methanol



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Ru-M paths



Without methanol

With methanol

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Ru-M paths



Without methanol

 ${\sf Ru}{\sf -}{\sf M}$ distances are longer and ${\sf Ru}{\sf O}_2$ is formed at high potentials

With methanol

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Ru-M paths



Without methanol

 ${\rm Ru}{\rm -}{\rm M}$ distances are longer and ${\rm RuO}_2$ is formed at high potentials

With methanol

Ru-M distances are shorter and remain the same at all potentials

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Ru-O/C paths



Without methanol

With methanol

"In situ Ru K-Edge x-ray absorption spectroscopy study of methanol oxidation mechanisms on model submonolayer Ru on Pt nanoparticle electrocatalyst," C.J. Pelliccione, E.V. Timofeeva, J.P. Katsoudas, and C.U. Segre, J. Phys. Chem. C 117, 18904 (2013).

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Ru-O/C paths



Without methanol

Above 375 mV Ru-O paths appear and total number of Ru-O neighbors increases to that of ${\rm RuO}_2$

With methanol

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Ru-O/C paths



Without methanol

Above 375 mV Ru-O paths appear and total number of Ru-O neighbors increases to that of ${\rm RuO}_2$

With methanol

Ru has one low Z neighbor at all potentials (carbon); a second above 175 mV (oxygen) with constant bond lengths and slightly increasing numbers

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Bi-functional mechanism





"In situ Ru K-Edge x-ray absorption spectroscopy study of methanol oxidation mechanisms on model submonolayer Ru on Pt nanoparticle electrocatalyst," C.J. Pelliccione, E.V. Timofeeva, J.P. Katsoudas, and C.U. Segre, J. Phys. Chem. C 117, 18904 (2013).

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V

• In situ He-filled box for non-aqueous battery experiments



- In situ He-filled box for non-aqueous battery experiments
- Anode materials measured include: Sn₃O₂(OH)₂, SnO₂, Sn, ZnO, MoO₂...





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- Anode materials measured include: Sn₃O₂(OH)₂, SnO₂, Sn, ZnO, MoO₂...
- Pouch cell simplifies experiment







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• MRCAT 10-ID beam line scans EXAFS spectrum in 2 minutes



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- MRCAT 10-ID beam line scans EXAFS spectrum in 2 minutes
- Focus on Sn nanoparticles which have rapid failure rate



- In situ He-filled box for non-aqueous battery experiments
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- MRCAT 10-ID beam line scans EXAFS spectrum in 2 minutes
- Focus on Sn nanoparticles which have rapid failure rate
- Develop modeled Sn-Li paths in $Sn_3O_2(OH)_2$ using 3 composite paths

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Initial in situ Sn-based anode EXAFS



 $Li_{22}Sn_5$ has 14 Sn-Li paths with distance of 3.4 Å or less. Model with three Sn-Li paths at "center of mass" locations

"In situ XAS study of the capacity fading mechanism in hybrid $Sn_3O_2(OH)_2$ /graphite battery anode nanomaterials," C.J. Pelliccione, E.V. Timofeeva, and C.U. Segre, *Chem. Mater.* **27**, 574-580 (2015).

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Initial in situ Sn-based anode EXAFS





 $Li_{22}Sn_5$ has 14 Sn-Li paths with distance of 3.4 Å or less. Model with three Sn-Li paths at "center of mass" locations



The number of Li near neighbors varies with lithiation and delithiation but shows how degradation progresses

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Sn nanoparticles – EXAFS versus potential





"Potential-resolved in situ x-ray absorption spectroscopy study of Sn and SnO₂ nanomaterial anodes for lithium-ion batteries," C.J. Pelliccione, E.V. Timofeeva, and C.U. Segre, J. Phys. Chem. C 120, 5331-5339 (2016).

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The Sn lithiation process



V

0.60V - Sn metal begins to break down and Li appears

 $0.45V-number of Li reaches 11 and stabilizes at near full <math display="inline">\rm Li_{22}Sn_5$

However, Sn fades rapidly due to electric conductivity loss. What can be improved?

"Potential-resolved in situ x-ray absorption spectroscopy study of Sn and SnO₂ nanomaterial anodes for lithium-ion batteries," C.J. Pelliccione, E.V. Timofeeva, and C.U. Segre, *J. Phys. Chem. C* **120**, 5331-5339 (2016).

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$Sn_4 P_3/graphite \ composite \ anode$

 Sn_4P_3 synthesized by high energy ball milling, then ball milled again with graphite to obtain composite

"In situ EXAFS-derived mechanism of highly reversible tin phosphide/graphite composite anode for Li-ion batteries," Y. Ding, Z. Li, E.V. Timofeeva, and C.U. Segre, Adv. Energy Mater. 8, 1702134 (2018).

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$\mathsf{Sn}_4\mathsf{P}_3/\mathsf{graphite}\ \mathsf{composite}\ \mathsf{anode}$

 Sn_4P_3 synthesized by high energy ball milling, then ball milled again with graphite to obtain composite







 Sn_4P_3 /graphite composite shows stable, reversible capacity of 610 mAh/g for 100 cycles at C/2 compared to rapidly fading pure Sn_4P_3 material.

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In situ EXAFS of Sn_4P_3 /graphite





"In situ EXAFS-derived mechanism of highly reversible tin phosphide/graphite composite anode for Li-ion batteries," Y. Ding, Z. Li, E.V. Timofeeva, and C.U. Segre, Adv. Energy Mater. 8, 1702134 (2018).

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In situ EXAFS of Sn₄P₃/graphite





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In situ EXAFS of Sn_4P_3 /graphite





Results for *in situ* coin cell are close to the capacity of the unmodified cell at C/4, indicating good reversibility by the 3^{rd} cycle.

"In situ EXAFS-derived mechanism of highly reversible tin phosphide/graphite composite anode for Li-ion batteries," Y. Ding, Z. Li, E.V. Timofeeva, and C.U. Segre, Adv. Energy Mater. 8, 1702134 (2018).

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By the third lithiation and third delithiation, the difference between pure Sn_4P_3 and the $Sn_4P_3/graphite$ composite is clear.

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 Sn_4P_3 /graphite composite

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 Sn_4P_3 /graphite composite

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Even at the 100^{th} delithiation, the Sn₄P₃/graphite composite measured *ex situ* is showing the same features as at the 3rd cycle.

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[&]quot;In situ EXAFS-derived mechanism of highly reversible tin phosphide/graphite composite anode for Li-ion batteries," Y. Ding, Z. Li, E.V. Timofeeva, and C.U. Segre, Adv. Energy Mater. 8, 1702134 (2018).

Example fits

Fit EXAFS for bond lengths and co-ordination numbers



The Sn-O peak at OCV is due to ball milling, which introduces oxygen.



"In situ EXAFS-derived mechanism of highly reversible tin phosphide/graphite composite anode for Li-ion batteries," Y. Ding, Z. Li, E.V. Timofeeva, and C.U. Segre, Adv. Energy Mater. 8, 1702134 (2018).

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Example fits

Fit EXAFS for bond lengths and coordination numbers



By the 3^{rd} lithiated state, the EXAFS is dominated by Sn-Li paths at 2.7 Å and 3.0 Å.



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Example fits





At the 3^{rd} delithiation, the Sn-P path reappears but at a shorter distance, in an amorphous SnP_x phase.



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Sn-Sn distance close to those of metallic Sn indicate the presence of small Sn clusters which may never fully lithiate

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Longer Sn-P distance characteristic of Sn_4P_3 is gone after initial conversion to the SnP_x amorphous phase is complete

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Sn-O distances remain constant, likely indicative of surface contamination

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Sn-O neighbors decrease quickly, remaining small and partially reversible up to 100 cycles

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The \sim 3.3 Sn-P neighbors in the delithiated state indicate a possibly tetrahedral Sn coordination in SnP_x

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Amorphous SnP_x fully formed at start of 3^{rd} cycle



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a. Sn lithiating

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Amorphous SnP_x fully formed at start of 3^{rd} cvcle



- a. Sn lithiating
- **b**. SnP_x all gone

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Third cycle dynamic snapshot



Amorphous SnP_x fully formed at start of 3^{rd} cvcle



- a. Sn lithiating
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 - f. Sn delithiated

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Photoemission is the complement to XAFS. It probes the filled states below the Fermi level



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The dispersion relation of electrons in a solid, $\mathcal{E}(\vec{q})$ can be probed by angle resolved photoemission since both the kinetic energy, \mathcal{E}_{kin} , and the angle, θ are measured





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$${\cal E}_{kin}, \ heta \ \longrightarrow \ {\cal E}(ec q)$$

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The work function, ϕ , is the minimum energy required to promote an electron from the top of the valence band at the Fermi energy, \mathcal{E}_F , to the vacuum energy, \mathcal{E}_V





With the incident photon energy, $\hbar\omega$, held constant, an analyzer is used to measure the kinetic energy, \mathcal{E}_{kin} , of the photoelectrons emitted from the surface of the sample





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the core states are used to fingerprint the chemical composition of the sample

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The electric field between the two hemispheres of radius R_1 and R_2 has a R^2 dependence from the center of the hemispheres





V

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Electrons with $\mathcal{E}_0,$ called the "pass energy", will follow a circular path of radius

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Electrons with different azimuthal exit angles ω will map to different positions on the 2D detector





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The total momentum of the photoelectron is calculated from the measured kinetic energy





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the electron dispersion curve can be fully mapped by sample rotations



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ARPES experimental data





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measurement of K edges of 3d elements, L edges of 5d elements, and M edges of 5f elements

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ability to measure bulk photoemission and buried interfaces as well as the surface

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