

Surface Science

Room 120 - Session SS+AMS-MoA

Surface Chemistry and Reactivity on Oxide Surfaces

Moderators: Ashleigh Baber, James Madison University, J. Anibal Boscoboinik, Center for Functional Nanomaterials, BNL

1:30pm SS+AMS-MoA-1 Dynamic Formation of Gem-Dicarbonyl on Rh Decorated Fe₃O₄(001), Jiří Pavelec, C. Wang, P. Sombut, L. Puntischer, M. Eder, Vienna University of Technology, Austria; Z. Jakub, CEITEC, Czechia; R. Bliem, Advanced Research Center for Nanolithography, Netherlands; M. Schmid, U. Diebold, Vienna University of Technology, Austria; C. Franchini, University of Vienna, Austria; M. Meier, G. Parkinson, Vienna University of Technology, Austria

Single atom catalysts (SACs) have the potential to reduce the amount of precious materials needed in catalytic reactions. Understanding and utilizing SACs requires studying the coordination of adatoms to their supports, as well as their coordination to reactants. This coordination can change during reactions, and intermediate dynamic steps may be invisible to conventional spectroscopy.

In this study [1], we employ scanning tunneling microscopy (STM) in combination with theory to investigate a model SAC: Rhodium decorated Fe₃O₄(001). Our results demonstrate that the formation of dicarbonyl on Rh₁ requires the initial presence of Rh₂ on the surface, a finding corroborated by detailed density functional theory (DFT) studies.

CO adsorption at Rh₁ sites at room temperature results exclusively in stable Rh₁CO monocarbonyls, as the Rh atom adapts its coordination to form a stable pseudo-square planar environment. Rh₁(CO)₂ gem-dicarbonyl species are also observed, but they form exclusively through the break up of Rh₂ dimers via an unstable Rh₂(CO)₃ intermediate. Identification of this intermediate step would be challenging without a multi-technique approach.

These results are compared to the Rh decorated TiO₂ model SAC using temperature-programmed desorption (TPD), infrared reflection absorption spectroscopy (IRAS) [2], nc-AFM, and X-ray photoelectron spectroscopy (XPS).

[1] Wang, C.; Sombut, P.; Puntischer, L.; Jakub, Z.; Meier, M.; Pavelec, J.; Bliem, R.; Schmid, M.; Diebold, U.; Franchini, C.; Parkinson, G. S., *Angewandte Chemie* **2024**, 63 (16).

[2] Rath D.; Mikerásek V., Wang C.; Eder M.; Schmid M.; Diebold U.; Parkinson G.S., Pavelec J. **2024**, *submitted*

1:45pm SS+AMS-MoA-2 Water-Gas Shift Reaction Mechanisms on Ligand Coordinated Pt Single Atom Catalyst: Insights from DFT & Microkinetics, Dave Austin, D. Le, T. Rahman, University of Central Florida

Hydrogen is a promising renewable and environmentally friendly fuel to meet future global energy needs. An important reaction to produce hydrogen is the water-gas shift (WGS) reaction, (CO+H₂O→CO₂+H₂; ΔH=-41.1 KJ/mol). Interest in the use of single-atom catalysts (SACs) for facilitating these reactions has grown. This project explores a new strategy that can create a metal-ligand coordinated SAC on metal oxide (Titanium oxide) support. 1,10-phenanthroline-5,6-dione (PDO), was chosen as the ligand for its oxidative potential for stabilizing metal cations in two bidentate sites. Our experimental collaborators Fereshteh Rezvani and Steve Tait from Indiana University were able to characterize the single-atom nature of the Pt with EXAFS, XPS, XRD, DRIFTS, and TEM. They evaluated for the WGS reaction, and it was discovered that Pt-ligand SAC supported on defective TiO₂ shows higher inherent catalytic activity than Pt NPs with significantly lower activation energy which is generally desirable for the redox mechanism.

First, the structure of the Pt-PDO molecule had to be determined, this is because the ligand has two bidentate sites. Experimental results that this complex has a ratio of 2:1 for the ligand to Pt, giving three different potential structures. After the molecule was determined, we had to understand the role of vacancies on the TiO₂ surface. The Pt-PDO complexes (all three configurations) were adsorbed onto to TiO₂ surface that was either clean, or had an oxygen, titanium, or oxygen and a titanium vacancy. We showed that the vacancies were important to activate the Pt-PDO complex, on the clean surface there is only van der Waals interaction between the surface and the complex. The electronic structure of these systems also shows the activation of the Pt atom from vacancies, as the Pt becomes more reactive and has its frontier orbitals pushed closer to the

Fermi level. The WGS reaction has two different proposed mechanisms. They are the Redox and the associative mechanisms. They differ by which point the reactants are adsorbed. These two mechanisms' pathways were simulated on the Pt-PDO complex and to understand the role of ligands the Redox mechanism was also studied on a single Pt atom. The DFT calculations confirmed that the redox mechanism has a lower energy barrier than the associative mechanism for Pt-ligand SAC. A microkinetic study was also performed to obtain the theoretical turnover frequency for all three reactions. The microkinetic study shows that the Redox mechanism has a higher turnover frequency than that of the associative and single Pt atom reactions.

2:00pm SS+AMS-MoA-3 Surface Chemistry and Catalysis of IrO₂(110), Jason Weaver, University of Florida; A. Asthagiri, Ohio State University; M. Kim, Yeungnam University, Republic of Korea; J. Jamir, C. Pope, University of Florida; J. Yun, Ohio State University; S. Ramasubramanian, University of Florida

INVITED

Developing more efficient catalytic processes to oxidize light-alkanes partially or completely is important for various applications, including power generation, exhaust gas remediation and chemical synthesis. In this talk, I will discuss investigations of alkane oxidation on the IrO₂(110) surface, and emphasize how mechanistic insights obtained from UHV surface science experiments and DFT calculations have been used to inform our understanding of rates and species coverages measured during alkane oxidation under catalytic conditions. I will discuss the development of a first-principles microkinetic model that accurately reproduces key aspects of the kinetics of methane oxidation on IrO₂(110) and identifies how different surface species, observed using operando spectroscopy, affect the catalytic kinetics. I will also discuss recent results which clarify how gaseous H₂O influences the catalytic oxidation of ethane on IrO₂(110) and the surface species that develop under reaction conditions. Our studies demonstrate how fundamental knowledge gained from surface science and DFT calculations can play a critical role in interpreting operando measurements and identifying the mechanisms of complex, catalytic reactions.

2:30pm SS+AMS-MoA-5 Room Temperature Activation of Methane and Its Dry Reforming by MgO Nanostructures Embedded in CuO_x on Cu(111), Areebin Islam, K. Reddy, Brookhaven National Laboratory; Y. Tian, Stony Brook University/Brookhaven National Laboratory; J. Rodriguez, Brookhaven National Laboratory

Natural gas, primarily methane, is valued for its versatility and potential in sustainable energy production through reforming or partial oxidation reactions. The investigation stems from the need for efficient technologies to utilize natural gas for sustainable syn gas or hydrogen production while simultaneously addressing carbon dioxide (CO₂) utilization and conversion challenges. MgO nanostructures show promise for methane activation due to their unique surface properties, while Cu-based catalysts are explored for selective methane oxidation at lower temperatures. This study investigates the growth and reactivity of MgO nanostructures on a Cu₂O/Cu(111) substrate, employing scanning tunneling microscopy (STM) and synchrotron-based ambient-pressure X-ray photoelectron spectroscopy (AP-XPS). Deposition of Mg atoms on the "29" structured copper oxide film induces oxygen transfer from the Cu₂O/Cu(111) substrate to the deposited Mg, forming MgO and CuO_x. Diverse morphologies are observed, including structured copper oxide films with embedded MgO clusters (1-3 Mg atoms) and randomly dispersed MgO nanoparticles. Reactivity studies reveal that MgO nanostructures smaller than 1 nm in width activate methane at room temperature, dissociating it into CH_x species. CO₂ dissociates into CO and C species instead of forming plain carbonates. The size and morphology of MgO nanostructures significantly influence their reactivity, enabling dry reforming of methane (MDR) by CO₂ into syn gas. Specifically, MgO nanostructures with a coverage of 0.04-0.11 ML activate methane at room temperature, leading to its dissociation into CH_x species. Additionally, smaller MgO clusters (0.2-0.5 nm in width, 0.4-0.6 Å in height) at lower coverages exhibit distinct reactivity towards CO₂, dissociating it into CO and C species. This investigation underscores the size- and morphology-dependent reactivity of MgO nanostructures, showcasing a behavior distinct from bulk MgO. The catalytic performance observed is attributed to the unique magnesium-copper interface, featuring multifunctional sites comprising magnesium cations, oxygen, and copper cations, which facilitate methane activation and drive the MDR processes forward at around 500K.

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2:45pm **SS+AMS-MoA-6 Mixed IrO₂/RuO₂(110) Thin Films: Distinct Surface Chemical Properties of the Single-Layer Oxides**, *Suriya Narayanan Ramasubramanian, C. Sudarshan, J. Shin, C. Lee*, University of Florida, Gainesville; *C. Plaisance*, Louisiana State University; *D. Hibbitts, J. Weaver*, University of Florida, Gainesville

Mixed metal-oxides of IrO₂ and RuO₂ have potential to serve as efficient catalysts for promoting the partial or complete oxidation of alkanes, due to the unusual ability of IrO₂ to activate light alkanes at low temperature as well as the possibility that the mixed oxides exhibit distinct surface chemical properties compared with the pure oxides. In this talk, I will discuss our recent studies of the growth and surface chemical properties of layered structures of IrO₂(110) and RuO₂(110) thin films as well as mixed IrO₂-RuO₂(110) films prepared in UHV. We find that single-layers (SL) of IrO₂(110) on RuO₂(110) and vice versa exhibit distinct binding properties toward adsorbed molecules compared with the corresponding multilayer (ML), bulk-like oxides. TPD shows that the binding of N₂ and O is stronger on SL-RuO₂(110) on ML-IrO₂(110) relative to ML-RuO₂(110), whereas these species bind more weakly on SL-IrO₂(110) on ML-RuO₂(110) relative to ML-IrO₂(110). These differences are especially pronounced for oxygen in that the binding energy of an adsorbed O-atom on top of a surface metal site increases in the order, SL-IrO₂ < ML-RuO₂ < ML-IrO₂ < SL-RuO₂, with the binding energy differing by ~50 kJ/mol between each single vs. multiple layer structure. I will discuss DFT calculations which show that these differences originate from a trans-ligand effect, wherein the bonding properties of surface metal atoms are strongly influenced by the bonding of sub-surface O-atoms to the second layer oxide. Lastly, I will discuss recent results showing that well-mixed Ir_xRu_yO₂(110) thin films can be generated in UHV and will discuss their surface chemical properties. The significant differences between the surface chemical properties of single vs. multiple layer oxide structures may have broad implications for understanding the catalytic behavior of mixed IrO₂/RuO₂ systems.

3:00pm **SS+AMS-MoA-7 Active Sites for Oxidation Reactions on Cu₂O Surfaces**, *Dario Stacchiola*, Brookhaven National Laboratory

Cu-based catalysts are active for partial and full oxidation reactions. Copper can be oxidized under moderate oxidant pressures and temperature to Cu₂O, and further to CuO under typical catalytic reaction conditions. We present here model systems using both copper oxide thin films and single crystals used to interrogate the effect of modifiers on the stability of exposed active Cu sites. *In situ* experiments allow the observation of dynamic processes and phases under reaction conditions.

References

- [1] "Oxidation of CO on a reconstructed Cu₂O surface", (submitted)
- [2] "Stabilization of Cu₂O through site-selective formation of a Co₂Cu hybrid single-atom catalyst", *Chem. Mat.* **34**,2313(2022)
- [3] "Potassium-Promoted Reduction of Cu₂O/Cu(111) by CO", *J. Phys. Chem. C* **123**, 8057–8066 (2019)
- [4] "Redox Properties of Cu₂O(100) and (111) Surfaces", *J. Phys. Chem. C* **122**, 28684–28691 (2018)

3:15pm **SS+AMS-MoA-8 Tracking Elementary Steps in Conversion of Carboxylic Acids on Single Crystalline and Nanofaceted TiO₂(101)**, *Xingyu Wang*, Pacific Northwest National Lab; *W. Debenedetti*, Los Alamos National Laboratory; *C. O'Connor*, Harvard University; *Z. Dohnalek, G. Kimmel*, Pacific Northwest National Lab

Ketonization of carboxylic acid used to be a method to produce acetone in industry. Recent interest has focused on this C-C coupling reaction due to its potential for upgrading biomass. The production of acetone from acetic acid was only observed in high pressure reactors on anatase nanoparticles. However, on anatase TiO₂(101) single crystals in ultra-high vacuum (UHV), acetone production from acetic acid has not been observed. This is an example of the material gap in surface science studies. The mechanism of ketonization is also under debate; the two most commonly proposed pathways are β-keto acid pathway and ketene pathway.

In this study, we introduced well-defined nanoparticles (NPs) with mostly (101) surfaces into a UHV chamber to elucidate the ketonization mechanism and bridge the material and pressure gaps in this system. A combined experimental approach of temperature programmed desorption (TPD), scanning tunneling microscopy (STM), X-ray photoelectron spectroscopy (XPS), and reflection absorption infrared spectroscopy (RAIRS) was used, along with theoretical studies. Our finding is that on single crystals, the ketene produced desorbs from the surface without encountering another acetate. In contrast, ketene desorbing from a given NP within a layer of NPs can subsequently react with an acetate on another

NP, leading to acetone production. To demonstrate this, we prepared three samples with varying thickness of anatase NP layers, with mostly (101) facets, in an UHV system. We then compared the reaction of acetic acid on these NP layers with its reaction on an anatase(101) single crystal. We found that the production of acetone starts from 10ML of acetic acid exposure, and the yield correlates with the depth of acetic acid absorption into the nanoparticle beds. Along with theoretical studies, we identified a mechanism through a key intermediate, α-enolate acetic acid, which forms through the reaction of gas phase ketene with surface-bound acetate species. Further studies, which involve dosing ketene through a homemade heated quartz tube ketene source onto an acetic acid pre-dosed single crystal surface, are currently underway to confirm this reaction mechanism.

3:30pm **SS+AMS-MoA-9 Developing First-principles Microkinetic Models for Selective Ethane Oxidation on Cl-substituted IrO₂(110)**, *Jungwon Yun*, The Ohio State University; *D. Bae, N. Park*, Yeungnam University, Republic of Korea; *J. Weaver*, University of Florida; *M. Kim*, Yeungnam University, Republic of Korea; *A. Asthagiri*, The Ohio State University

In this study, we investigated the role of Cl doping on ethylene selectivity for ethane oxidation on IrO₂(110) using a combination of density functional theory (DFT) and microkinetic modeling (MKM). Catalytic oxidative dehydrogenation (ODH) of ethane is an attractive route to produce value-added products such as ethylene. Our previous research demonstrated that stoichiometric IrO₂(110) exhibits significant potential for ethylene production at low temperature (~ 400 K) due to the low activation energy for initial C-H bond cleavage of light alkanes. Temperature programmed reaction spectroscopy (TPRS) experiments showed that surface HO groups promote ethylene selectivity in ethane ODH. Given Cl is isoelectronic to surface HO groups and has been experimentally shown to be able to substitute bridge O atoms on RuO₂(110) surfaces, we explored Cl substitution effects on ethylene selectivity on IrO₂(110). DFT calculations indicated that the presence of bridge Cl on the IrO₂(110) surface destabilizes adjacent adsorbed ethane and ethylene but has minimal impact on the reaction barriers of C₂H_x species with adjacent oxygen. This suggests that limited Cl substitution would still allow the IrO₂(110) surface to convert C₂H₆ to C₂H₄, but subsequently start to constrain further dehydrogenation and oxidation steps due to the lack of bridge O atoms. A DFT-based MKM was developed to understand the relationship between product yields (C₂H₆, C₂H₄, CO/CO_x) on degree of Cl substitution. The MKM modeled TPRS confirms that ethylene yield is increased by Cl substitution but eventually reaches a maximum where there is a drop due to the deactivation of the surface. This maximum in ethylene yield is dependent on initial C₂H₆ coverage since dehydrogenative oxidation steps produce surface HO groups that, when combined with bridge Cl, deactivate the surface. We will discuss ongoing work to extend the MKM simulations to reaction conditions where activity and selectivity are examined as a function of partial pressures of C₂H₆ and O₂ along with percent Cl substitution.

4:00pm **SS+AMS-MoA-11 Small Alcohol Reactivity Over TiO₂/Au(111) Inverse Model Catalysts**, *Ashleigh Baber*, James Madison University

Gold-based catalysts have received tremendous attention as supports and nanoparticles for heterogeneous catalysis, in part due to the ability of nanoscale Au to catalyze reactions at low temperatures in oxidative environments. Surface defects are known active sites for low temperature Au chemistry, so a full understanding of the interplay between intermolecular interactions and surface morphology is essential to an advanced understanding of catalytic behavior and efficiency. Our undergraduate research lab uses ultrahigh vacuum temperature programmed desorption (UHV-TPD) to investigate the fundamental interactions between small alcohols on Au(111) and the reactivity of TiO₂/Au(111) inverse model catalysts on small alcohol redox behavior. In a systematic study to better understand the adsorption and intermolecular behavior of small alcohols (C₁-C₄) on Au(111) defect sites, coverage studies of methanol, ethanol, 1-propanol, 1-butanol, 2-butanol, and isobutanol have been conducted on Au(111). These small alcohols molecularly adsorb on the Au(111) surface and high resolution experiments reveal distinct terrace, step edge, and kink adsorption features for each molecule. The desorption energy of small primary alcohols was shown to trend linearly with increasing C₁-C₄ carbon chain length, indicating that the H-bonded molecular packing of 1-butanol resembles that of methanol, ethanol, and 1-propanol, while isobutanol and 2-butanol deviate from the trend. These energy insights are particularly interesting when studying the redox behavior of small alcohols over TiO₂/Au(111). Depending on the surface preparation conditions, Au(111) supported TiO₂ nanoparticles react with

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small alcohols to form either reduced and oxidized products. The reactivity of the surface for ethanol oxidation was altered by controlling the oxidation state of TiO_x ($x < 2$) and coverage of TiO_2 . Low coverages of fully oxidized TiO_2 nanoparticles on Au(111) are active for the selective oxidation of ethanol to form acetaldehyde, but not all small alcohols behave similarly.

4:15pm **SS+AMS-MoA-12 Partial Chlorination of IrO_2 (110) for Selective Ethane Chemistry**, Connor Pope, University of Florida, Gainesville; J. Yun, Ohio State University; R. Reddy, J. Jamir, University of Florida, Gainesville; M. Kim, Yeungnam University, Republic of Korea; A. Asthagiri, Ohio State University; J. Weaver, University of Florida, Gainesville

Developing more efficient catalytic processes to convert ethane to ethylene is important for improving hydrocarbon-to-chemicals processing and transitioning to carbon-neutral technologies. Our prior work demonstrates that C_2H_6 dehydrogenation on the IrO_2 (110) surface produces C_2H_4 between ~350 and 450 K during temperature programmed reaction spectroscopy (TPRS), and that the C_2H_4 selectivity can be increased by pre-hydrogenating the oxide surface to deactivate a fraction of the surface oxygen sites. In this talk, I will discuss recent work in which we controllably replaced surface O-atoms of IrO_2 (110) with Cl-atoms through the oxidation of gaseous HCl in ultrahigh vacuum. We find that the stepwise adsorption of HCl on IrO_2 (110) with heating to 650 K causes H_2O desorption, and generates Cl atoms on bridging and on-top sites in about a 1:1 ratio, with the total Cl coverage saturating near 0.7 ML. Measurements using TPRS and XPS further demonstrate that the partitioning of Cl-atoms between bridging and on-top sites can be altered by exposing the surfaces to reducing vs. oxidizing conditions. Lastly, I will discuss how partial chlorination affects the surface reactivity toward CO and C_2H_6 , and produces a nearly two-fold increase in the selectivity of C_2H_6 dehydrogenation to C_2H_4 during TPRS. Our findings demonstrate the potential of controlled surface deactivation for improving the selectivity of IrO_2 (110) for partial alkane oxidation.

4:30pm **SS+AMS-MoA-13 Insights into CO_2 Hydrogenation on the InO_x/Cu (111) and InO_x/Au (111) Surfaces: Surface Electronic Structure and Reaction Mechanistic Studies**, Prabhakar Reddy Kasala, J. Rodriguez, Brookhaven National Laboratory

In CO_2 hydrogenation, In_2O_3 catalysts are known for their higher CH_3OH selectivity, primarily attributed to their oxygen vacancies. Subsequent studies explored depositing metals on In_2O_3 to enhance CO_2 conversion and oxygen vacancy formation.¹⁻² Besides metal supported on In_2O_3 -based catalysts, intermetallic In-M (M= Pd, Cu) compounds have also shown good performance for CO_2 hydrogenation to methanol synthesis.³⁻⁴ Despite extensive research on metal (M) supported In_2O_3 catalysts, the role of In-M alloy and M/ In_2O_3 interfaces in CO_2 activation and methanol selectivity remains unclear. Our surface electronic structural studies using APXPS reveal that during CO_2 hydrogenation, very low coverage (~0.035 ML) InO_x/Cu (111) undergoes structural changes at/above 500 K, forming a Cu-In intermetallic alloy at the surface. Further increase in Indium coverage (0.325 ML) results in InO_x/Cu (111) at the surface, while the interface remains an In-Cu alloy. Carbon 1s core level spectra of the InO_x/Cu (111) regions of the surface under 1 Torr of $\text{CO}_2 + \text{H}_2$ (1:3 ratio) show the formate (HCOO^*) and H_xCO^* species, with H_xCO having the higher concentration at 500 K in 0.035 ML, while the 0.325 ML Indium oxide has shown the carbonate (CO_3^*) at 300 K and decreased with an increase in temperature to 500 K. Our results clearly demonstrate the promotional effects of In_2O_3 for CO_2 hydrogenation. Furthermore, we extended our studies on InO_x/Au (111) and $\text{InO}_x/\text{TiO}_2$ (001) surfaces to understand the role of Oxide/Metal, Alloy, Oxide/Oxide roles in methanol selectivity. Our preliminary STM and XPS results on InO_x/Au (111) established the preparation of In/Au (111) and InO_x/Au (111) surfaces. We are making progress on indium-based catalysts for CO_2 hydrogenation to establish the role of possible alloys/intermetallic and metal-support interfaces in CO_2 activation and CO_2 hydrogenation to methanol under reaction conditions.

References:

1. Cao et. al., *ACS Catal.* 2021, 11, 1780-1786
2. Rui et. al., *ACS Catal.* 2020, 10, 11307-11317
3. Shi et. al., *J. Catal.* 2019, 379, 78-89
4. Chen et. al., *ACS Catal.* 2019, 9, 8785-8797

4:45pm **SS+AMS-MoA-14 Structural-Electronic Property Evolution of LiCoO_2 (001) Under Varied Oxygen Chemical Potentials**, Yuchen Niu, J. Reutt-Robey, University of Maryland College Park

Since its discovery as an intercalation Li-ion battery electrode in 1980, LiCoO_2 remains a popular cathode material for portable devices. Its surfaces also provide an unexplored opportunity to tune the structure and charge-

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transport properties for emergent electronic applications. In this study, we report on the evolution of LiCoO_2 (001) surfaces, specifically their structural and electronic property response to processing under low/high O_2 chemical potential extremes via scanning probe microscopy (SPM), low energy electron diffraction (LEED), X-ray photoelectron spectroscopy (XPS) and Raman spectroscopy.

Under a traditional UHV regimen at low O_2 chemical potentials, LiCoO_2 (100) persistently displays a diffused (1x1) LEED pattern after many cycles of Ar^+ sputtering- thermal annealing, suggestive of nanoscale disorder without major atomic surface reconstruction. UHV-STM imaging reveals the singular surface terminated by shallow grains of ~2 nm width, which coarsen to ~30 nm grains under prolonged processing. Atomically resolved images of local regions show ($\sqrt{7} \times \sqrt{7}$) R 19.1° ordering, which could be attributed to local Li ordering on Li-terminated layers. The electronic properties of this surface are further mapped with UHV scanning tunneling spectroscopy. Electronic band gaps derived from I-V spectroscopy are reported. This is corroborated by in situ XPS measurements of the surface chemical composition and valence band structures during the Ar^+ sputtering- thermal annealing cycles.

Under high O_2 chemical potentials (160~ 760 torr O_2), dramatic changes in surface structures are observed, indicating more efficient surface mass transport. In contrast to the nanograin features observed at low O_2 chemical potentials, high O_2 chemical potential treatments generate expansive atomically flat (100) terraces of ~ 1 μm width, step edges and quasi-hexagonal islands. Partial surface decomposition to Co_3O_4 due to Li loss is also observed.

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