Iridium(111), Iridium(110), and Ruthenium(0001) Single Crystals as Model Catalysts for the Oxygen Evolution Reaction: Insights into the Electrochemical Oxide Formation and Electrocatalytic Activity

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We report a comparative study on the influence of generic electrochemical activation–oxidation protocols on the resulting surface oxides of Ir(111) and (110) and Ru(0001) single crystals and their electrocatalytic reactivity for the oxygen evolution reaction. Well-defined single-crystal electrodes were prepared in a custom-made chamber that combines inductive thermal annealing and electrochemistry. The clean surfaces were analyzed for their electrocatalytic oxygen evolution activities and oxidation behavior. Three different oxidation protocols were used, which revealed a strong activity dependence on the duration and upper potential limit of the electrochemical oxidation. The resulting changes of the surface were followed by using cyclic voltammetry and impedance spectroscopy. Important differences between the two faces of Ir in terms of surface morphology of the formed oxide were identified, which allowed us to draw conclusions for preferable crystal faces in nanoparticle catalysts.

Introduction

With regard to the increasing worldwide need for energy, the provision of sustainable energies has become one of the greatest challenges of the 21st century. The intermittency of renewable sources, such as wind and solar power, however, overshadows their advantages of harmlessness with minimal impact on the environment and opens the need for energy storage technologies. The electrochemical splitting of water into oxygen and hydrogen using polymer electrolyte membrane (PEM) electrolyzers is considered to be a viable technology for an appropriate energy storage system.[1] However, the efficiency of water electrolyzers is considered to be a viable technology for an appropriate energy storage system.[2] However, the efficiency of water electrolysis is limited by the sluggish kinetics of the oxygen evolution reaction (OER), and thus, the development and optimization of new catalysts for the OER is of great importance for a practical grid-scale application of PEM electrolyzers.[3]

Commonly applied PEM OER catalysts constitute complex systems, which impede the analysis of the activity-determining-material properties. Hence, a well-defined model system is required to obtain fundamental knowledge about the OER.[5] Well-defined single-crystalline electrodes are suitable model catalysts to study the influence of different facets separately to further optimize the design of new electrocatalytic materials, which we apply in this study.

Here, we discuss the structural effects of distinct crystal facets of oxidized single-crystalline Ru and Ir electrodes on their catalytic oxygen evolution performance. We establish a relationship between their OER activities and electrochemical surface characteristics as a function of three basic types of oxidative surface–activation protocols: i) direct (Di), ii) dynamic (Dy), and iii) direct dynamic oxidation (Dd). The directly oxidized single crystals form their active oxides during the oxygen evolution process. The increase of the upper turning potential through dynamic oxidation results in a similar active IrOx species for both Ir facets. The application of Dd oxidation generates a much more active RuOx@Ru(0001) interface compared to the Dy oxidation protocol, which is contrasted by IrOx@Ir(110). The Dd oxidation of Ir(111), however, features an OER performance comparable to that in the Dy oxidation. Our study provides new fundamental insight into the sensitivity of
the catalytic OER performance of well-defined electro-oxidized metal–metal oxide surfaces to the nature of applied interfacial potential protocols and gives practical guidance for the design of suitable activation procedures of Ru and Ir OER catalysts.

Results and Discussion

We selected three types of potentiodynamic surface-activation protocols, namely, i) Di, ii) Dy, and iii) Dd, to study the interfacial voltammetry and the electrocatalytic behavior of electrochemically formed surface oxides on well-defined single-crystal facets of Ir and Ru. Detailed procedures are provided in the Experimental Section.

We first focused on the cyclic voltammetric (CV) behavior of each single crystal during each individual activation procedure. The recorded profiles during the Dy protocol of all three facets are shown in Figure 1. Starting at a constant lower turning potential of 0.05 V, the upper turning potential was successively raised in 100 mV steps from 0.8 to 1.4 V to monitor the changes of the CV features by the formation of surface oxide species. The facets display distinctly different behavior.

The CVs of Ir(111) shown in Figure 1 a are characterized by late-onset oxidation shown by peak broadening and a shift to more positive potentials of the hydrogen underpotential desorption (HUPD) feature only after the upper turning potential (E2) was increased above 1.2 V. The inset shows the emergence of the formation of hydrous iridium(IV) oxide and its reduction to an iridium(III) oxide species, which is distinguished by a large reduction peak at 0.9 V that only appears after E2 > 1.2 V as well. At a scanning speed of 50 mV s⁻¹ up to a potential of 1.2 V, the Ir(111) catalyst is rather stable before it loses its well-ordered structure through oxidation in the following scans. Equivalent to Ir(111), the HUPD peak at 0.07 V of Ir(110) (Figure 1 b) decreases, but in contrast to that of Ir(111) the decrease starts from E2 = 0.9 V and changes gradually with an increased upper potential limit, which demonstrates the loss of its single-crystalline character. However, we observe an increase in the current density of its shoulder at approximately 0.15 V. In contrast to the closed structure of Ir(111), the oxide redox peaks shown in Figure 1 b (regions 2 and 3) exhibit distinctly higher current densities, which indicate that the structure of the (110) crystal surface (which possibly also includes the missing row reconstructed surface) facilitates the incorporation of oxygen in the metal structure as it offers better access to the interlattice sites and, therefore, a deeper penetration of the electrolyte into the metal. We also notice a shift of the reduction peak of the OH adsorption to more negative potentials, which suggests the high stability of its formed oxide.

The Ir(110) surface undergoes stronger changes during oxidation that start at lower potentials than that of the Ir(111) surface, which suggests a distinct oxidation process. The stable voltammetric response of the Ru(0 0 0 1) surface is shown in Figure 1 c. Two additional oxidation features at 0.9 and 1.1 V are observed that do not change with the upper turning potential. The reduction peaks at 0.48, 0.42, and 0.25 V increase in intensity, which reflects the greater amount of oxide formed.
at higher upper potential limits, but no irreversible change is visible. An additional redox feature at 0.08 V is observed at the highest potential limits. This feature is attributed to an oxide species formed irreversibly. A detailed description of the redox features is presented in Figure 2. The Ru(0001) surface does not undergo irreversible changes during most of the Dy protocol. Only a small portion of irreversible oxide formation takes place in the chosen potential regime. Scans to intermediate values of $E_2$ have been omitted for clarity.

The initial activity of the three single-crystal surfaces after the three different oxidation protocols under comparative consideration are displayed in Figure 3. The first and second scan into the OER region for the Di method are compared in Figure 3a. The Ir(110) surface shows a significantly lower overpotential during the first scan than the Ir(111) surface. In the second scan, the Ir(110) surface remains almost unchanged and is then nearly matched by the (111) surface. As iridium oxide is expected to constitute the active species in oxygen evolution, this suggests that the closely packed (111) surface is more stable under oxidative conditions and takes longer to fully oxidize into the active surface Ir oxide.

The more open structure of the Ir(110) surface seems to complete the surface oxide formation during the first scan, hence the larger hysteresis. The (110) surface might facilitate the incorporation of oxygen in the metal structure as it offers better access to the inner lattice sites. The ratio of the atoms per unit area of Ir(111) ($1.57 \times 10^{15}$ cm$^{-2}$) and Ir(110) ($1.92 \times 10^{15}$ cm$^{-2}$) is 1.2, which is significantly lower than the ratio of their current densities at a fixed potential of 1.53 V (9.6). Therefore, the difference in the OER activity of both Ir faces cannot be an effect of the number of active sites alone. The fact that both facets converge in the CVs of the activated materials may further suggest that their active catalyst phase is quite similar in structure and composition. Ru(0001) exhibited the highest observed OER activity in this comparison but showed a slightly increased overpotential in the second scan. Something similar was found for polycrystalline materials. Similar OER scans obtained after application of the Dy protocol (Figure 1) are compared in Figure 3b. Herein we see that the initial activity of Ir(111) and (110) matches and does not change in the second scan. Hence, this 21 cycle oxidation protocol up to a potential of 1.4 V is sufficient to oxidize both surfaces to identical activity. For Ru the effect of Dy is a weak increase of its overpotential. To elucidate whether the number of cycles or the upper turning potential is the determining factor, a third set of experiments in which the Dd protocol was applied was conducted, wherein the surfaces were first oxidized in the potential range of 0.05–1.4 V. The resulting OER activities are shown in Figure 3c. The most striking difference is the higher overpotential for Ru(0001), which decreases over the following cycles to reach a value similar to that after the other protocols. This behavior may be explained by the well-known formation of RuO$_4$. The dissolution of electrode material apparently is accompanied by a roughening, which leads to an increase in the active area. After three oxidative cycles, the Ir(111) surface still exhibits a slightly larger overpotential than the corresponding

![Figure 2](image-url)

**Figure 2.** Effect of oxidation during OER on features in the Faradaic potential range of the surface voltammograms of a) Ir(111), b) Ir(110), and c) Ru(0001).

![Figure 3](image-url)

**Figure 3.** First and second OER scans measured at 5 mV s$^{-1}$ in 0.05 M H$_2$SO$_4$ of a) Di, b) Dy, and c) Dd samples of Ir(111) (black), Ir(110) (red), and Ru(0001) (green). The 25th scan and the SPV scans are shown in c) instead of the second scan.
(110) surface, which proves that not only upper potential but the cycle number (or oxidation time) is important to form catalytically active surface oxides. As stated previously, the Ir atoms in the (110) surface oxidize readily, and the formed oxide displays a good catalytic activity. After 25 scans, the Ir(110) surface still exhibits a slightly lower overpotential than the (111) surface but they are almost the same. However, the decrease in activity is more pronounced on the (110) face, which indicates that the (111) face exhibits a more stable and closed oxide surface. In the stepped potential voltammetry (SPV) measurements, Ir(111) is slightly more active than (110) but the activities are very close, which shows that both oxidized Ir surfaces form a chemically comparable, active oxide layer.

In this comparative oxidative study, the Ru catalyst revealed a higher overpotential in the second scan than that in the first scan during direct and dynamic oxidation. This is not the expected observation if we consider a possible active site increase by corrosion of Ru during the OER. However, it has been shown that under acidic conditions the formation of a more stable anhydrous ruthenium oxide layer, also referred to as passivation of Ru, occurs, which may result in the increasing overpotential in the second scan. Although there is a higher dissolution contribution to the current in the first OER scan, the possible formation of a passivating oxide layer reduces its contribution in the following scan. In the further course of oxidation, more active RuO$_x$ sites are produced through oxide growth into the metal to result in an activity increase during the following 25 scans.

To assess the effect of repeated voltammetric excursions into the potential region of the OER on the resulting surface redox electrochemistry, CVs were recorded before, during, and after the OER scans shown in Figure 3. These CVs were conducted in the potential range of 0.05–0.8 or 0.05–1.0 V for Ir and Ru, respectively, to probe only those potential regions that feature reversible Faradaic reactions and to avoid irreversible oxide formation. This choice of CV potential ranges ensures that only the stable forms of the active oxide species are probed.

Ir(111) changes mainly in the HUPD region (Figure 2 a). Here, the main peak for the stripping of adsorbed hydrogen atoms diminishes with consecutive cycles into the OER region, which is attributed to oxide formation. Thus, after 25 cycles, the surface appears to be oxidized almost completely. For Ir nanoparticles, the disappearance of the HUPD is a sign of fully oxidized particles, in the sense that no Ir metal participates in the reaction. Moreover, peak broadening and a shift of the peak center to more positive potentials occurred, which indicates the loss of the highly ordered structure during this oxidation (cf. Figure S4). An initial increase in capacitive current density is visible but remains constant for subsequent scans, which indicates that the surface roughness increases upon oxidation, but once formed, the oxide surface remained unaltered. In the potential region around 0.7 V a slight increase in current density is observed that may be caused by OH adsorption or the beginning of Ir oxidation. In contrast, the features of Ir(110) showed more distinct changes (Figure 2b). The HUPD main peak (1) decreases, whereas the shoulder (2) increases and subsequently stays constant during the stability test (Figure S4a). A decrease and a shift to more positive potentials is visible for the HUPD main peak of Ir(110) after 25 scans into the OER region. This observation demonstrates the loss of the single-crystalline character. Two chemically different Ir species exist on the (110) face as indicated by the presence of two HUPD peaks. The peak at 0.07 V can be assigned to outer Ir atoms, and the shoulder at 0.15 V is caused by subjacent Ir atoms (for further explanation, see Figure S4b). The main HUPD peak at 0.07 V decreases gradually as the corresponding Ir atoms are oxidized in the first place and no longer show metallic character. Once the subjacent atoms are oxidized they may permit the ready access of the electrolyte to the first layer(s) of metal underneath the oxide. The additional contribution of these atoms may lead to an increase in the current density of the shoulder. As discussed for the (111) surface, the capacitive current increases initially but stays constant after the first oxidation. At 0.65 V, a redox couple for OH ad-/desorption (3) becomes visible after the initial oxidation. The final shape of the CV resembles the typical shape of oxidized polycrystalline bulk Ir electrodes. If we consider these oxidized polycrystalline Ir electrodes, the presence of an HUPD peak is explained by a porous or proton-conducting oxide layer that allows the buried interface (Ir metal in this case) to participate in the reaction. As iridium oxides formed electrochemically are known to be highly hydrated, a proton transfer mechanism through the thin oxide layer seems possible. A comparison of the oxidation behavior of the two investigated Ir faces with the findings mentioned above reveals the plausibility of a closed oxide layer formed on the Ir(111) surface with no participation of the buried interface in the surface reactions, whereas on Ir(110) the metal interface still contributes to the redox features after extensive oxidation during the OER, which thus renders it comparable to polycrystalline Ir. This finding dismisses our earlier hypothesis of a possibly identical catalytically active oxide phase on Ir(111) and Ir(110); instead, it suggests the formation of two distinctly different metal oxide surface structures formed on the investigated crystal planes. Ru(0001) shows an earlier onset for the exchange of adsorbed hydrogen sulfate with hydroxide ions (1) (0.3 instead of 0.5 V) and an additional redox couple for oxide formation (2) (0.8 V) after the initial oxidation (Figure 2c). Also, the oxide reduction peak (3) and its shoulder (4) (at 0.48 and 0.42 V, respectively) merge to form a single broad peak. The surface OH $^\text{•−}$/HSO$_4^\text{−}$ exchange peak (5) at 0.25 V also shifts to slightly more negative potentials, which reflects the stronger binding of oxide species after oxidation. Another redox couple emerges at approximately 0.08 V (6), which is attributed to further oxide/hydroxide reduction. After 25 scans, the capacitive current increased threefold and, although all the features are still visible, they are relatively smaller, which is caused by severe surface roughening and a surface that approaches a polycrystalline structure.

The measured total anodic charge Q between 0.4 and 1.4 V of the investigated metal surfaces at the pristine stage, after 25 cycles of OER, and after the SPV is shown in Figure 4. Q is repeatedly interpreted as a relative measure of the active
oxide surface area of Ru and Ir oxides. Both Ir surfaces show an initial increase of $Q$ upon oxidation, which stays constant afterwards. This behavior indicates that for a given, fully oxidized Ir face, the value of $Q$ does not depend on the applied oxidation procedure. Thereby, the formed oxides are highly stable and protect the metal below from oxidation efficiently. A comparison with the similar activity results shown in Figure 3 implies a similar active surface species. Notably, the increase on Ir(111) is significantly larger than that on Ir(110), which indicates that Ir(111) undergoes more severe changes during oxidation and can be explained by the more open structure of the (110) surface that allows easier oxidation and has a larger surface area from the start. However, the surface charge of Ru increases steadily, which is in line with the findings from the CV measurements as a strong indication of surface roughening caused by Ru corrosion in form of RuO$_4$. Hence, in the case of Ru, the real surface area increases and so does the active surface area. Conclusively, the activity of Ru(0001) is related to its anodic charge, which is influenced strongly by the pretreatment.

For a deeper understanding of the different oxides formed on Ir(111) and (110), impedance spectroscopy measurements were conducted during the SPV steps. The resulting internal resistances were used to correct for the iR-drop of the potential during the SPV. The potential dependent change of the charge transfer resistance $R_{ct}$ is shown in Figure 5 together with the corresponding Tafel plot that shows the logarithm of the geometric current density ($\log(j_{geo})$) on the second y axis. The corresponding equivalent circuit diagram is described in Figure S6.

The $R_{ct}$ obtained from fitting the semicircle of each impedance spectrum decreases for both Ir catalysts with increasing oxidation potentials. This semicircle behavior characteristic of progressive oxidation is in line with the Tafel behavior. Here, the electron transfer rate into the electrode is controlled by the electrochemical reaction. The decrease in charge transfer resistance indicates a decreased effect on the activation energy with the increasing overpotential.

Two distinct linear regions can be distinguished in the Tafel plots for both orientations. The transition potential between the low and high Tafel slope region is 1.58 V, at which the transient charge was determined on the pristine catalysts (initial), on the oxidized catalysts after OER (25 scans), and again after SPV.

![Figure 4. Total anodic charge $Q$ measured by CV. Inset: CV between 0.4–1.4 V with a scan rate of 50 mV s$^{-1}$. The charge was determined on the pristine catalysts (initial), on the oxidized catalysts after OER (25 scans), and again after SPV.](image)

![Figure 5. Potential dependent change of Tafel slope (right y axis) and $R_{ct}$ (left y axis) measured by impedance spectroscopy after each SPV step for a) Ir(111) and b) Ir(110); 200 kHz $> f >$ 5 Hz; U = ±10 mV sinus.](image)
sition of IrVII to IrVI takes place according to the Pourbaix dia-
gram. For Ir(111) the Tafel slope increases from 59 to
89 mV dec⁻¹ (Figure 5a), and the Tafel slope increases from 64
to 83 mV dec⁻¹ for Ir(110) (Figure 5b) in line with values re-
ported previously. The kink in the Tafel slope can be attribu-
ted to a change in the OER kinetics, for example, the rate-de-
termining step.

Interestingly, despite the different nature of the catalytically
active oxides (see Figure 3a and 3b) we observe similar behav-
ior of both oxidized Ir surfaces in the Tafel slope and
impedance spectroscopy.

Conclusions

We have prepared single-crystal electrodes of Ir(111), Ir(110),
and Ru(0001) in a modified and improved inductive electro-
chemical single-crystal chamber. This improved custom-made
in situ single-crystal cell design expands the functionalities re-
ported previously in studies on Pt-based fuel cell catalysts with
the high-temperature annealing of oxygen evolution reaction
(OER) catalysts. The well-defined and clean surfaces were ex-
amined with respect to their electrochemical oxidation behav-
ior and resulting OER activity. In this context, three different
oxidation protocols were applied. The results of this analysis
showed that single-crystalline Ru, even though it is distinctly
more active than Ir, is highly unstable as the formation of solu-
ble RuO₂ leads to huge losses of electrode material and sur-
face roughening. If we compare the oxidized samples of Ir,
Ir(110) is distinguished by a lower overpotential than Ir(111) in
the first scans. Nevertheless, in the following scans Ir(111) ap-
proaches the OER activity of Ir(110). The initially higher activity
of Ir(110) is attributed to its more open and accessible surface,
which thus allows faster oxidation. The activity equalization
and the comparable behavior in the impedance spectroscopy
speak in favor of a chemically similar surface species on the
fully oxidized Ir catalysts. In contrast to the observed OER ac-
tivity, the nature of the underlying metal surface orientation
appears to influence the structure and morphology of the gen-
erated oxide strongly, which leads to distinctly differing redox
features. A significant difference is the missing hydrogen un-
derpotential desorption peak on the (111) crystal. As pure IrO₂
does not show hydrogen underpotential desorption, the for-
mation of different oxide structures is possible. While the oxi-
dized (110) layer permits access to the underlying metal, the
oxidized (111) surface seems to prevent electrocatalytic
penetration and thus hydrogen adsorption.

Our study provides new insights into the sensitivity of the
catalytic OER performance of well-defined electro-oxidized
metal–metal oxide surfaces to the nature of applied activation
protocols.

For Ru, the results of previous studies could be confirmed and,
despite its superior activity, strong dissolution and rough-
ening render it unfeasible as a single-component OER catalyst.

Depending on the desired properties of the OER catalysts,
this study can provide insight on possible surface-activation
routes for Ir catalysts. A constant or transient anodic oxidation
without further reduction leads to a thin and less hydrated
oxide, whereas the application of an oxidation protocol with
cycling between oxidizing and reducing potentials leads to
thicker, hydrated oxides with a possible 3D reaction volume.

The different Ir surface layers that are oxidized influence the
oxide morphology and thus lead to possible suggestions for
shape-controlled nanostructures with preferential surface
planes. Depending on the application of the catalyst, a porous
or dense oxide could be preferred. Ir(111) gives a dense oxide
that shows no sign of metallic Ir features, which could be real-
ized with octahedral structures as it is known for Pt-based
nanoparticles. Fully oxidized Ir(110) still exhibits a hydrogen
underpotential desorption from the metallic Ir in the buried in-
terface, which allows a larger reaction regime in the porous
oxide and makes this kind of oxide suitable as a possible bi-
functional catalyst with Ir features, which could be realized in
rod-like nanostructures.

Experimental Section

Single-crystal catalysts of Ir(111), Ir(110), and Ru(0001) (MaTecK,
purity 99.999%, oriented to <0.1.>) cylinders with a diameter of
10 mm and a height of 4 mm) were mounted with Ir wires (Alfa
Aesar, ϕ = 0.25 mm, 99.9%) to the crystal holder after cleaning in
Milli-Q water.

Before each electrochemical experiment, well-defined and clean
single-crystal surfaces were prepared by inductive heating in an
electrochemical single-crystal chamber constructed according to
Ref. [16] with modifications (Figure S1). The gas-tight cell consists
of two compartments: the electrochemical compartment at the
bottom and the inductive compartment at the top. The thermal
 treatment of the Ir single crystals was performed at (1900 + 40) °C
in a H₂ (4%)/Ar (purity 99.999%) atmosphere for 2 min, and the
Ru single crystal was annealed at (1200 ± 40) °C in an Ar atmos-
phere for 4 min repeatedly until the electrochemical characteriza-
tion of the surface layers, which means that the charge for the
HUPD was consistent with calculated values (Table S1 and Fig-
ure S4).

All electrochemical measurements were performed in the inductive
electrochemical single-crystal setup specifically designed for this
purpose. This setup includes an electrochemical cell at the bottom
with a Luggin capillary (Figures S1 and S2). A saturated mercury/mer-
cury sulfate (HMS) reference electrode was used and calibrated
frequently against a reversible hydrogen electrode (RHE) in the
same electrolyte at RT. All potentials provided here were converted
and refer to the RHE unless specified otherwise. A 0.05 M H₂SO₄ so-
lution was used as the electrolyte and was prepared by the dilu-
tion of 95% H₂SO₄ (Suprapure, Carl Roth) with Milli-Q water
(18 MΩcm at RT). The electrolyte was degassed with Ar (15 min)
before each measurement and after extensive oxygen evolution
during the OER measurements. All electrochemical measurements
were performed by using a potentialstat (SP-240, BioLogic, France).

The pristine single-crystalline surfaces obtained after thermal treat-
ment were subject to three different oxidation protocols: Di, Dy,
and Dd. Di consists of a simple anodic scan from the immersion
potential at 0.4 to 1.23 V followed by two cycles of quasi-stationary
CV (5 mV s⁻¹) up to a current density of 1 mA cm⁻². Thereby, the
impact of surface oxide formation on the OER activity can be
traced. During Dy, the upper turning potential of a series of CVs
was increased successively cycle by cycle (Figure 1). Starting at 0.8
and 1.0 V for Ir and Ru, respectively, the upper turning potentials

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were increased to 1.4 V with an increment of 0.1 V. Subsequently, the resulting OER performance was measured. To distinguish between the influence of the upper turning potential and the number of cycles on the formation of the active oxide, Dd was applied. Distinct from Dy, the single-crystal surfaces were oxidized by three CV scans in the potential range of 0.05–1.4 V followed by 25 consecutive voltammetric OER performance cycles. To determine the resulting surface redox electrochemistry of the Dy protocol, CVs were conducted before, during, and after the OER scans. This oxidation protocol further includes quasi-stationary scans by using SPV, which consists of a series of anodic steps of 20 mV starting from 0.1 mA cm$^{-2}$. Each potential was held for 2 min to attain a quasi-steady-state current density before impedance spectroscopy was measured.

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Two faced: The morphological influence of Ir(111), Ir(110), and Ru(0001) single crystals on oxide formation and the resulting oxygen evolution reaction activity is presented. Precise pretreatment control reveals distinct surface oxides on the studied single-crystalline model catalyst. These results provide guidelines for enhanced catalyst design.